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MAGNETOSTRICTIVE MATERIAL

This invention relates to the production of materials with useful magnetostrictive properties.

Magnetostriction is the property which relates magnetic characteristics of a body of material to a change of the shape of the material. The property is seen in the change in size of bodies of certain materials when the magnetic environment changes or the change in magnetic characteristic when a force is applied to such a body to change its shape. Magnetostriction is a dimensionless quantity represented by the magnetostriction constant λ_s, relating magnetisation and shape change and in the SI system of units useful values are some tens or hundreds of parts per million, particularly for use in sensors and transducers. For such uses a high magnetomechanical coupling factor is desirable. Also "soft" magnetic properties are generally preferred.

While thin film amorphous alloys and magnetic multilayers individually provide some of the required properties there is still a strong need for a significant improvement in the properties available and for materials which exhibit a useful combination of such properties.

It is an object of the invention to provide materials with improved properties and methods of production of such materials.

According to the invention there is provided a magnetic material of at least two component parts arranged to have respective structures which mutually are not homogenous, the structure of one part cooperating with the structure of the other to assist the magnetostrictive behaviour of the material.

The cooperation may be a pseudo free surface on a part.

The cooperation may reduce restriction of the local moment of a part.

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The cooperation may be the structure of one part altering the atomic spacing of the other. Preferably the alteration is an increase from the usual spacing. The structure may be the absence of a regular lattice.

There may be an interface region in one component where said first and second components are contiguous. There may be an interface region where the layers are contiguous. The modification may be in the interface region. Where the interface region is in an elemental component the lattice structure may be one other than the usual one for the elemental component. Where the interface region is in a component which is at least partly an alloy the alloy may have a ratio other than the usual one for said alloy.

A third component layer may be overlaid on the second and a lattice structure of said third layer may be modified in its turn. Further component layers may be overlaid with or without lattice structure modification.

A layer may have a lattice structure modified by an overlaid layer, at least contiguous to the overlaid layer.

Each of the component layers may have an inherent lattice structure and modification may be that at least one inherent structure is modified by another contiguous inherent structure.

The modification may be that the second lattice, at least where contiguous to the first component layer, adopts at least approximately the first lattice form.

One of the first and second component layers may be a magnetic component and the other may be magnetic or a non-magnetic component. While each of the first and second layers will generally be different elements or alloys it is also possible magnetic and non-magnetic forms of one component may be used.

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The magnetic component may be chosen from the group of ferromagnetic elements of at least iron, nickel, cobalt, gadolinium and terbium.

The non-magnetic component may be chosen from aluminium, magnesium, gold and silver and silicon oxide.

Preferably there are many first and second layers in the material forming a multilayer material. Generally there will be alternating said first and second layers. One first layer and the overlaid second layer may be identified as a bilayer. A first and second layer may have a third layer associated therewith as a trilayer and may further have a fourth layer associated therewith as a quadlayer. In general the material form can be expressed as of M multilayers each having N component layers. Typically N will be between two and five while M will have a value of a few tens, typically 20 to 50.

There may be a substrate on which the multilayer material is built up. The material may remain on the substrate for use or be released therefrom, for example by chemical means. The substrate may be glass or mica or KAPTON (RTM) or other suitable material. The magnetic material may be prepared in the place of eventual use, for example by deposition of successive component layers on a machine part or like object. There may be more than 30 bilayers.

Each layer of a bilayer may be at least a few nanometres thick, that is a few tens of atoms thick, and may be up to ten or more nanometres thick. It is believed that a well-defined boundary is useful where the second layer is overlaid on the first. This will produce a stress-free strain in the overlaid layer.

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One form of the material described above may prepared by a method including overlaying the second component on the first, providing a well-defined boundary where the second component is contiguous with the first, and causing or producing a stress-free strain in the overlaid component, at least adjacent the first component.

Another form of the material described above may prepared by a method including overlaying the second component on the first, permitting or causing the second component structure to be influenced by the first to produce said cooperation.

According to a particular aspect of the invention there is provided a magnetic material of a first component layer and a second component layer overlaid on the first, there being a lattice structure in at least one of said layers and in which magnetic material at least one lattice structure is modified where said first and second component layers are contiguous whereby the magnetostrictive property of the material is enhanced.

There may be an interface region where the layers are contiguous. The modification may be in the interface region. Where the interface region is in an elemental layer the lattice structure in at least said region may be one other than the usual one for the layer element.

According to the invention there is provided a method of producing a multilayer magnetic material including depositing a first component, causing or permitting a second component to be progressively overlaid on the first, controlling the depositing of the first component and the overlaying of the second component to produce respective different structures and causing or permitting the structures to cooperate to assist the magnetostrictive behaviour of the material.

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The method may include providing a first component layer of a first lattice structure, causing or permitting a second component layer of a second lattice structure to be progessively overlaid on the first, controlling the overlaying of the second component so that at least one component layer in a region contiguous to the interface of the layers has a lattice structure modified by the overlaying action.

The method may include overlaying by sputtering. The method may include overlaying substantially atom-by-atom. The method may include sputtering from a target which may be of one element or of two or more elements to enable the deposit of an alloy.

The method may include causing or permitting the respective different structures to be distinct and form an interface which provides a pseudo free surface.

A multilayer material as described above has an M/H loop characteristic which is altered by distorting the material, for example by bending.

According to a particular aspect of the invention there is provided a multiple multilayer magnetic material as described above in which the overlaid second layer is covered by an insulating layer, to enhance the frequency response.

According to the invention there is provided a magnetic material arrangement including a magnetic material as described above and means to cooperate with the material to convey to an external connection a response of the material to an external action.

The response may be a change of permeability or dimension and the external action may be the stressing of the material. The action may be flexure of a support for the material or an ambient pressure change, or an external magnetic field.

The arrangement may form part of an accelerometer, a torque sensor or like device.

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Embodiments of the invention will now be described with reference to the following examples and drawings, in which:

Figure 1 shows in schematic form a representation of a lattice structure of a multilayer material according to the invention,

Figure 2 shows in schematic form a representation of another lattice structure of a multilayer material according to the invention,

Figure 3 shows M/H characteristics of a material according to the invention,

Figures 4 and 5 show in schematic form a portion of a multilayer material, after deposition and after subsequent annealing respectively,

Figures 6 to 9 show plots of characteristics of materials embodying the invention,

Figure 6 shows magnetostriction constant of Fe/Ag multilayers (S1) versus 1/(modulation period),

Figure 7 shows magnetostriction constant of Fe-Co/Ag multilayers versus 1/(modulation period) calculated using E_f from both the volume average and the vibrating reed correction from equation 5, (squares and circles respectively). Also shown are the effective magnetostriction constants of the alloy layers alone, using the volume average E_f method (triangles),

Figure 8 shows magnetostriction constant of Fe-Co/Ag multilayers versus 1/(modulation period),

a) t_{Ag} = 2 nm (S4),

b) t_{Ag} = 5 nm (S3),

c) t_{Ag} = 5 nm, t_{anneal} = 30 minutes (S2),

Figure 9 shows magnetostriction constant of Fe-Co layers within Fe-Co/Ag multilayers versus 1/(modulation period), (a), (b) and (c) as in Figure 8, and

Figure 10 is a table of materials exemplifying the invention.

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Various proposals for multilayer materials having magnetic properties have been made, for example de Wit, Witmer and Dirne (Advanced Materials, Vol. 3 (1991) No. 7/8 pp 356 to 360) and Zeper, van Kesteren and Carcia (Advanced Materials, Vol. 3 (1991) No. 7/8 pp 397-399). In the first of these (de Wit, etc) a material with enhanced saturation magnetisation and relative permeability but minimal magnetostriction, specifically for a video recorder read/write head, is described. To achieve this a very small grain size is sought for the magnetic material layer (iron, grain size below 10 nanometres) and to produce such a grain size the iron layers are around 10 nanometres thick. The layers are separated by thinner layers of another ferromagnetic material, specifically an iron/chromium/boron layer. This layer needs to be at least two nanometres thick to prevent the grains in one iron layer linking with those in another iron layer by columnar growth and specifically epitaxial growth is not desired. In the second (Zeper et al) a magneto optical recording material is described, for enhanced recording density, which has adequate Kerr rotation and low enough Curie temperature while having a preferred magnetisation direction perpendicular to the material layer. This preferred direction only occurs with cobalt/platinum or cobalt/palladium multilayers when the cobalt layers are less than some 0.8 to 1.2 nanometres. The thickness of the non-magnetic but magnetically polarisable platinum or palladium layer is set by the required balance of Kerr effect and Curie temperature and for platinum is about 0.9 nanometres with a 0.4 nanometre cobalt layer. The cobalt layer is about two atom layers thick so that the required magnetic anisotropy is not reduced by "bulk" atoms between the surface layers. In Zuberek, Szymczak, Krishnan and Tessier (Journal de Physique, Vol. 12, No. 9, Colloque C8, December 1988, pp 1761 to 1762) it is suggested that a "bilayer" of evaporated component materials depends for

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effectiveness on the thinness of a nickel layer. In Dirne, Tolboom, de Wit and Witmer (J. Magn. Magn. Mat., No. 83, (1990) pp 399 to 400) the possibility of interface mixing in Fe/Co multilayers is discussed and seen as a disadvantage. Techniques to avoid this disadvantage are suggested.

Multilayers of the present invention do not follow the techniques of the above proposals. The present invention provides in one aspect that a layer of a first component overlaid on another layer, of a second component, is caused in a region contiguous with the other layer to adopt a structure other than that of the lattice which it would normally adopt. In this way, which can be described as lattice mismatch, a stress-free strain can be produced in at least the contiguous region of the overlaid layer. A result of this stress-free strain is an enhanced magnetostriction constant, λ_s.

Typically such layers of a first material overlaid on the second would be arranged as several bilayers on a substrate. Each layer of a bilayer would be some five to fifteen nanometres thick and deposited by sputtering so that atoms being deposited arrive at a rate slow enough to be able to be influenced in deposit position by the existing lattice of the second component layer or that of the first-deposited overlaid first component atoms. Care is needed to avoid building stress into the layers by the thermal effects of sputtering. Some ten to fifty bilayers can be deposited. The overlaid component applied adjacent to the second component lattice is conveniently caused to adopt a lattice spacing appropriate to that of the second component. This will cause a stress-free strain in at least the contiguous region of the overlaid component. The stress-free strain will be best exhibited if there is a sharp boundary between the layers.

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If required a pre-stress can be built in to the eventual material by, for example, keeping the substrate hot during deposition of components so that the substrate contracts on cooling. Also a magnetic bias can be built in by including a layer of permanently magnetisable component for example as or immediately above the substrate. A buffer substrate can also be used but care must be taken that the buffer transmits any strain that is applied for action by or on the magnetostrictive material. An inert or insulating layer may be applied to protect the material. Films may be grown under the influence of an imposed magnetic field to enhance the desired effect.

Figure 1 represents in a schematic manner the lattice at and near the surface of the second component layer and the lattice in the contiguous region of the overlaid component layer. In the Figure overlaid component atoms are indicated by the smaller, crossed, circles and the second layer component atoms by the more widely spaced larger, open, circles.

To investigate the magnetostrictive properties further examples have been prepared. Typically these are a number of alternate layers of magnesium and iron, each a few nanometres thick and having a few tens of bilayers. The layers are not necessarily of the same thickness. The layers are deposited by sputtering from alternately exposed sources using conventional techniques. The preparation is arranged to ensure that bulk effects are not significant. The effect of stress on the film on the shape of the magnetisation/field (M/H) loop was also examined. A small amount of distortion, by bending the specimens, changed the loop shape by a detectable amount, increasing the value of M at lower values of H compared with the loop for the unstressed specimens.

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Other structures may be used to achieve the invention. Certain combinations of overlaid component layers will produce a degree of alloying and this in turn can produce "dislocations" in an atomic level structure which would otherwise be adopted. Provided one component layer is magnetic the other can be an insulator as the merging of the layers where they are contiguous can alter the spacing of the magnetic component layer lattice and thus usefully alter the magnetostrictive property. Figure 2 shows in sketch form this action where atoms of a magnetic component, identified at m, are displaced by the presence of atoms of another component, which may be non-magnetic even an insulator, identifed at i.

The present invention provides in another aspect an interface effect which can be a relatively sharp lattice mismatch as the overlaid components cannot easily mix or in a yet further aspect a smoother mismatch which may be an intermetallic region as overlaid components merge. Component layers need not be deposited separately, the rate of a first layer may be reduced while the rate of a second layer is increased to take over.

An insulating layer, for example silicon oxide, can be introduced to produce an improvement in performance, especially an improved high frequency performance. The insulating layer thickness will be dependent on the frequency at which the device is to operate but will also be dependent on the effect of the relative thickness of the insulating layer and the bilayer on the propagation velocities in the insulating and magnetic components. From these considerations it is possible to derive suitable thickness relationships for a particular application of the multilayer material. In general the insulating layer thickness will be several times that of the individual magnetic component layer. In this way

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magnetostrictive materials with several tri- or quad- layers can be built up, of the general form of L multilayers each having N component layers. At least one of the N component layers will be a magnetic, generally a ferromagnetic, substance such as iron, nickel, cobalt, gadolinium or terbium while another will be a non-magnetic substance and may be a metal or an insulator, examples including aluminium, magnesium gold and silver and silicon oxide. If N is more than two one of the non-magnetic layers can be a metal and another an insulator. A further magnetic component layer may be included. A magneto-electric transducer can be formed by placing structures as exemplified above onto an electrostrictive structure such as PVDF, the dimensional changes in the magnetic structures imposing a strain on the electrostrictive structure, producing an electrical signal related to the action on the magneto strictive structure.

Further examples of the invention will now be described.

Multilayers are grown using an rf glow discharge sputtering system in a sputter-up configuration with three 15 cm water-cooled magnetron target electrodes on a carousel, and a water-cooled substrate platen electrode which allows the substrate to be pre-sputtered. The substrate platen also has a heater capable of temperatures up to 600°C. The target-substrate distance was set at 60 mm, with an argon plasma pressure of 5 mtorr. The sputtering equipment is a NORDIKO machine. The deposition rates were calibrated using a "Talystep" stylus based film thickness measurement instrument. Structural characterisation was also done using X-ray diffraction.

The substrates were glass and Kapton (Registered Trade Mark) (polyimide). The substrate for SAMR measurements (see below) was 25 mm thick Kapton, which was thin enough and had a low enough Young's modulus to avoid overly constraining the films while still being able to support them.

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Fe/Ag multilayers were grown with 50 repeats (i.e. 50 bilayers).

Fe-Co/Ag multilayers of 25 repeats (25 bilayers) were grown. The layers of Fe-Co alloy in the Fe-Co/Ag multilayers were produced by growing sandwiches of Fe/Co/Fe between the silver layers (Figure 4). The multilayer was subsequently annealed to allow interdiffusion of the Fe and Co to form a layer of alloy of thickness t(Co)+2t(Fe) (the Ag is immiscible with both) (Figure 5). The heat treatment was done in a non-inductively wound tube furnace, with the sample laid flat between two glass slides in a vacuum provided by a rotatry pump and nitrogen trap to eliminate oxidation. The anneal time and temperature were 30 minutes and 290°C respectively.

A series (S1) of five Fe/Ag multilayers was grown where t(Fe)=t(Ag)=1,2,3,4,5 nm.

A series (S2) of Fe/Co/Fe/Ag (i.e. Fe-Co/Ag) multilayers was grown where the thickness of the Co layer was twice that of each of the Fe layers so that the total thickness was the same for both, resulting in a post-anneal alloy of a nominal composition of $Fe_{50}Co_{50}$. The thickness of this layer was varied from 4 to 40 nm and t(Ag) was set at 5 nm. These were subsequently annealed in vacuum for 30 minutes at 290°C. Two other Fe/Co/Fe/Ag (i.e. Fe-Co/Ag) series were grown with silver layer thicknesses of 2 (S4) and 5 (S3) nm. The anneal times were longer for the films with thicker Fe/Co layers because of the greater degree of interdiffusion required. The anneal times (at 300°C) were 10, 20, 30 and 50 minutes for alloy layer thicknesses 4, 6, 8, 12 nm respectively.

As the layer thicknesses are decreased the volume fraction of the interface regions is increased. This increases the proportion of the multilayer material which is formed by atoms at or near the surface of a layer. The local moment is increased in the vicinity of a free surface so the local moment effect is enhanced as the interface volume fraction is increased. The texture of a layer is also significant and its

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retention can be enhanced by depositing the alloy directly and avoiding or reducing the annealing step. This will also encourage growth of the alloy in the [110] direction normal to the layer.

The coercive fields of Fe-Co/Ag multilayers are between 1 and 2 kAm^{-1}, reducing with layer thickness. Generally, the annealing rendered the multilayers magnetically softer. Also by annealing the multilayers were stress-relieved and the iron and cobalt layers were caused to mix more thoroughly. The interfaces with silver were probably sharpened, since silver is immiscible with iron and with cobalt. For the thinnest iron and cobalt layers (D=9nm, i.e. t_{Co}=2nm) the region between silver layers would have been almost completely alloyed as-deposited [Dirne and Denissen, 1989]. Annealing this multilayer increased (approximately doubling) its coercive field in distinction to the other multilayers, and it became impossible to measure the magnetostriction. This is considered below.

The length of an anneal required to achieve complete alloying at a given temperature will depend on the thickness of the component layers. Thus it is possible that the anneals did not completely alloy the layers, especially in the thicker multilayers, and that they were longer than required for the thinner multilayers. The optimum heat treatment may not have been used.

Magnetostriction measurements were made using the Small Angle Magnetisation Rotation method proposed by Norita et al. [1980] and widely used on amorphous ribbons.

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The basic principle of the method is as follows: the sample to be measured is saturated in the longitudinal direction (z) by a large dc field. A smaller ac field of frequency ω acts in the transverse direction (y). This results in a small degree of rotation of the macroscopic moment direction about the longitudinal direction. If a stress, σ, is also applied to the sample along this direction, the angle of moment rotation is given by

$$\theta = \frac{\mu_o M_s H_y}{H_z + H_d + H_\sigma} \qquad (1)$$

where H_d is the demagnetising field due to the sample shape, and H_σ is the stress-induced anisotropy field. This is given by

$$H_\sigma = \frac{3\lambda_s \sigma}{\mu_o M_s} \qquad (2)$$

The sample is mounted within a search coil whose axis is in the longitudinal direction. Thus a rotation of the magnetisation about this axis results in an induced ac voltage across the search coil with frequency 2ω, $V_{2\omega}$.

A change in the stress on a magnetostrictive material effects a change in the stress induced anisotropy field. In the case of a positively magnetostrictive material, a tensile stress tends to make the stress axis magnetically easier, so aligning the moments towards this axis. This would cause the angle of moment rotation to decrease, so decreasing the search coil output voltage.

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This change in output voltage can be compensated by adjustment of the dc field, H_z. The change in H_z is then equal to the change in H_σ. Thus, the magnetostriction constant, λ_s, is generally determined by plotting the change in H_z for a series of applied stresses, so that (where moment rotation is small)

$$\lambda_s = \frac{\mu_o M_s dH_z}{3 \; d\sigma_a} \Big| V_{2\omega}, H_y \; . \qquad (3)$$

The multilayers are too thin to be self supporting and need to be measured while still on their substrates. Although these can have elastic moduli far lower than the multilayers, they are much thicker and so accommodate a significant proportion of the stress, so it is necessary to correct for their presence.

This correction can be made by assuming that the multilayer is strongly adhered to the substrate so that they strain by the same amount and that the Young's modulus, E, of the whole is equal to the volume average of the component layers and the substrate. This assumes that the reed as a whole (i.e. substrate + multilayer) is effectively homogeneous. In this case the magnetostriction is given by

$$\lambda_s = \frac{\mu_o M_s}{3} \left[\frac{E_{av} A_{f+s}}{E_f} \right] \frac{dH_z}{d\sigma_a} \Big| V_{2\omega}, H_y \; . \qquad (4)$$

The same method of correction was also used by Wenda et al. [1987] on measurements of FeB and FeSiB single films sputtered onto polyester substrates.

Measurements of multilayer samples were taken on two slightly different SAMR apparatus arrangements. The dc field was applied using a solenoid, while the ac field was applied with a pair of Helmholtz coils. The sample was placed within a search coil (4000 turns) coaxially with the solenoid, in the centre of the Helmholtz coils. In the second configuration, a second, "bucking", coil was also present and was backed off

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against the search coil to cancel out any induced voltages common to both, i.e. due to sources other than the sample magnetisation.

The sample was clamped at both ends. The clamp at one end was fixed while that at the opposite end was connected to a cable which was loaded with known weights. The dc field was determined from the product of the current and the number of turns per unit length in the solenoid.

The values of B_s were obtained from literature, namely 2.0T for iron and 2.3T for $Fe_{50}Co_{50}$ alloy. The value of B_s for the whole multilayer was simply B_s for the relevant magnetic layer multiplied by its volume fraction.

The Young's modulus of the multilayers was measured using the vibrating reed method, which is fully described in Berry and Pritchet [1975]. It is based on the measurement of the resonant frequencies of various modes of cantilever vibration, f_n, of reeds clamped at one end.

Reeds were cut from the multilayer using a sharp blade and had free lengths and widths of approximately 13-20 mm and 1 mm respectively.

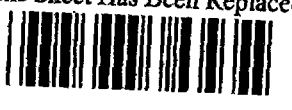
The value of E for the multilayer can be obtained from the value for the reed from the expression (from Berry and Pritchet):

$$\frac{f^2_{reed}}{f^2_{substrate}} = 1 + \frac{t}{d}\left[\frac{3E_f}{E_s} - \frac{\rho_f}{\rho_s}\right], \qquad (5)$$

where the subscripts f and s refer to the multilayer and substrate respectively, and t and d are the multilayer and reed thicknesses (t«d).

The value of λ_s for the multilayer was calculated from equation 4. In the case of the Fe-Co/Ag multilayers (t(Co)=2t(Fe)), two data sets are presented, corresponding to the two methods of calculating E_f. For the Fe/Ag data, only the values of λ_s derived from the assumption of a volume average E_f are shown.

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The data for λ_s is shown versus the reciprocal of the modulation period, D, in Figure 6. At 1/D=0 the value of λ_s should be that for bulk iron. The bulk value of λ_s of polycrystalline iron is -7 ppm, although this becomes more positive as it becomes more textured, so that perfectly textured polycrystalline iron with the [110] direction normal to the film surface has a value of λ_s of approximately 14 ppm. In practice, the measured value of λ_s for a single iron film will generally lie between these two values. As a check a thick (0.8 μm) iron film was deposited onto each side of a Kapton susbtrate (1.6 μm total). λ_s was measured to be -3.0 ppm.

As D decreases so that the volume fraction of the Fe/Ag interface layers increases, it is found that λ_s becomes more positive. Hence the presence of silver is seen to enhance the value of λ_s in iron, as expected.

A series of multilayers was grown with layers of Fe and Co of equal thickness (5 nm) with silver layers of varying thickness. Because there was approximately twice the amount of iron as cobalt in the alloy layer after annealing, it is probable that there was a composition gradient across the layer, with a corresponding variation in λ_s. The λ_s data from this series of multilayers did not show any systematic trend and there was a wide scatter in the data and is therefore not shown here.

The λ_s data for the second series of multilayers is shown for these films versus the reciprocal of the modulation period in Figure 7. Also shown is the effective λ_s of the magnetostrictive material alone, derived via the data assuming a volume average Young's modulus. There are pairs of data points obtained via the Berry and Pritchet E_f correction method for most samples, corresponding to two values of E_f obtained from the third and fourth modes of reed vibration. There are two samples measured with t(alloy)=30 nm since two sections

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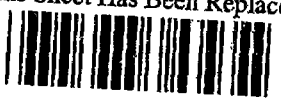
were taken from the same substrate and were annealed independently. However, E_{reed} for this sample was only measured using a reed from one of the anneals, so the pair of points from this sample with the higher λ_s values may be subject to large error. This figure illustrates the difference in the data resulting from the two methods of correction of the Young's modulus.

Before anneal, most samples were found to have small negative magnetostriction (generally smaller than -5 ppm), and large positive λ_s after anneal confirming that alloying of the component TM layers had alloyed. The sample with t(alloy)=4 nm gave a large positive λ_s before annealing, indicating that the iron and cobalt layers had intermixed to a high degree. It is suggested in Dirne and Denissen [1989] that Fe-Co alloys form at interfaces during deposition, with thicknesses of approximately 1 nm This means that approximately 50% of the Fe-Co in this sample was intermixed before anneal. Unfortunately, no SAMR response could be obtained from the sample after anneal. As stated earlier, this was accompanied by a remarkable increase in coercive field. It may be that since the layers were so thin, the anneal caused disruption of the coherency of the multilayer.

From the volume average data, it is seen that λ_s of the multilayer increases to approximately 20 ppm as the alloy thickness is reduced to 8 nm, equivalent to a value of λ_s of about 36 ppm for the alloy layers.

The magnetostriction of all three series (S2, S3, S4) of the Fe-Co/Ag multilayers were measured and are shown in Figure 8. These values were calculated using the volume average Young's moduli. The corresponding values of λ_s for the alloy alone are shown in Figure 9. A clear trend is seen for the series of multilayers (S4) with t_{Ag}=2 nm, where λ_s decreases with decreasing alloy layer thickness. The data for the series of multilayers with t_{Ag}=5 nm also show some

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reduction of λ_s with t_{Fe-Co}, but in Figure 8, the values of λ_s for the alloys are approximately constant (within error), indicating that the reduction in λ_s for the multilayer is due to loading by the silver layers. The reduction in λ_s for the t_{Ag} data set cannot be accounted for in this way.

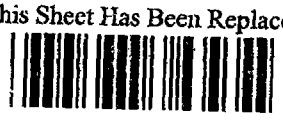
The decrease of λ_s of the thicker multilayers from the series (S2) is thought to be due to the iron and cobalt layers not fully intermixing during the half-hour anneal. It should be noted that the values of λ_s for the two multilayers comprising layers of 2 nm Fe, 4 nm Co and 5 nm Ag, annealed at approximately 300°C for 30 minutes were measured to be the same within error. This adds confidence to the repeatability and accuracy of the data. If intermixing is poor composition gradients can arise.

The value of λ_s of the alloy layer was measured to be approximately 35±3 ppm. This compares with approximately 80 ppm for the bulk. A multilayer of Fe/Co was deposited without silver layers, and was annealed nominally to form a single layer (0.5 μm total thickness) of Fe-Co alloy, T_a=300°C, t_a=30 minutes. The value of λ_s of the film was measured to be 43 ppm. A further heat treatment of 30 minutes at 300°C was found to cause an increase in λ_s to approximately 65 ppm.

The magnetostriction constant obtained from SAMR values depends on the Young's modulus of the reed and also on that of the film only. Discrepancies have arisen between the values of E_{film} corrected from E_{reed} from two different methods of obtaining this correction. The method assuming a volume average of the moduli is thought to be the more reliable.

The values of λ_s of the Fe/Co/Fe/Ag multilayers were small and negative, as would be expected from an average of Fe and Co. After anneal, the λ_s values were large and positive, indicating that the Fe and Co had alloyed to a large extent. Annealing also had the effect of making the films softer, predominantly due to relief of internal stresses. Values of H_c of the post-anneal samples were typically of the order of 1kA/m.

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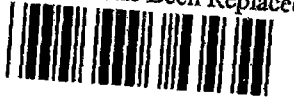
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Figure 3 shows the effect of stress on the M/H loop for one of the multilayers from series S4. The specific one used is that where n = 3. The multilayer had 25 bilayers, each deposited as 3 nm Fe/6 nm Co/3 nm Fe/2 nm Ag, supported on a 25 micrometre Kapton layer. Annealing was 50 minutes at 300°C under vacuum. The dotted line is the M/H characteristic at stress $\sigma = 0$. When a stress of 110 MPa is applied the shape and size of the M/H loop changes very significantly, shown in the solid line. M is in arbitrary units.

In the case of the Fe/Ag multilayers, reduction in the thickness of the layers (i.e. an increase in the volume fraction of the interface regions) has the effect of making the magnetostriction more positive. The measured values of λ_s in this study increased from approximately -0.3 ppm for 5 nm Fe/5 nm Ag to approximately +3 ppm for 1 nm Fe/1 nm Ag. This is consistent with an increase in the local magnetic moment of iron at the Fe-Ag interfaces.

In the case of the Fe-Co multilayers, λ_s was seen to decrease with decreasing modulation period, where this was shown to be due to mechanical loading by the silver layers, where t_{Ag}=5 nm. When this correction was made, the value of λ_{Fe-Co} were found to be approximately 35 ppm, which is considerably lower than those of bulk Fe-Co alloys. Non-optimal heat treatments are thought to contribute to the reduction in the values, as indicated by the decrease in λ_s for the thicker multilayers annealed for 30 minutes. A definite decrease in λ_s with decreasing alloy layer thickness was seen for the multilayers where t_{Ag}=2 nm, which cannot be accounted for by the loading by the silver layers. The reason for this is not clear. It is possible that the silver layers are so thin that the alloy layers have percolated through, especially where these are appreciably thicker than the silver, so that the parameters of the multilayer approach those of a bulk material. It may be that inclusion of silver in the multilayer causes a reduction in λ_{Fe-Co}.

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It is seen from the above that thickness and crystallographic texturing of the alloy layer and silver layer loading all can affect the value of λ_s. Those skilled in the art will be able to proceed on the basis of the techniques set out above to produce multilayers with significant values of λ_s using appropriate component layers and dimensions.

Some of the above examples show that the technique of causing a magnetic component layer to adopt, in a surface region at least, a lattice structure other than the one it would normally adopt produces a magnetic material which exhibits a non-zero magnetostrictive coefficient, albeit at a coercivity more appropriate to a "hard" magnetic material than a "soft" one, but noting that this parameter was not controlled.

Others of the above examples show that the technique of causing a sharp lattice mismatch or smoother mismatch intermetallic region in overlaid component layers produces a magnetic material which exhibits a non-zero magnetostrictive coefficient. In particular a sharp lattice mismatch, such as occurs with immiscible component layers, could produce a pseudo free surface. The term pseudo free surface is used to describe the arrangement where the surface of a thin layer can emulate a free surface although another layer is contiguous.

The magnetic material can be used in sensing devices, such as pressure gauges, accelerometers and torque sensors, by attaching the material to a structure or by depositing the material in the place where it is to be used.

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CLAIMS

1. A magnetic material of at least two component parts arranged to have respective structures which mutually are not homogenous, the structure of one part cooperating with the structure of the other to assist the magnetostrictive behaviour of the material.

2. A material according to Claim 1 in which the cooperation is the structure of one part altering the atomic spacing of the other part.

3. A material according to Claim 2 in which the alteration is an increase from the usual atomic spacing.

4. A material according to Claim 1 in which the structure of at least one part is the absence of a regular lattice.

5. A material according to Claim 1 in which there are contiguous component parts and in which the cooperation is a pseudo free surface on at least one said part.

6. A material according to Claim 1 in which there are contiguous component parts and in which the cooperation reduces restriction of the local moment otherwise achieved by said contiguous component parts.

7. A material according to Claim 1 in which there is an interface region in one component where said first and second components are contiguous.

8. A material according to Claim 7 in which the interface region is in a component which is at least partly an alloy and the alloy has a ratio other than the usual one for said alloy.

9. A material according to Claim 1 in which one of the first and second components is a magnetic material and the other is a magnetic or a non-magnetic material.

10. A material according to Claim 1 in which the first and second components are different elements or alloys or are magnetic and non-magnetic forms of one element or alloy.

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11. A material according to Claim 10 in which a magnetic component is chosen from the group of ferromagnetic elements of at least iron, nickel, cobalt, gadolinium and terbium.

12. A material according to Claim 10 in which a component is chosen from the group of elements of at least aluminium, magnesium, silver and gold and silicon dioxide.

13. A material according to Claim 1 including many first and second components as layers.

14. A material according to Claim 22 including alternating said first and second components.

15. A material according to Claim 1 including a first and second component having a third component associated therewith as a trilayer.

16. A material according to Claim 15 having a fourth component associated therewith as a quadlayer.

17. A material according to Claim 1 having a material form expressed as M multilayers each having N components as layers, where M is between two and five while M has a value of a few tens, typically twenty to fifty.

18. A material according to Claim 1 including a substrate on which the material is built up.

19. A material according to Claim 18 in which the material remains on the substrate for use or is released therefrom, for example by chemical means.

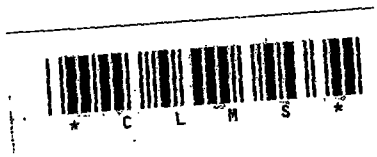
20. A material according to Claim 18 in which the substrate is glass or mica or KAPTON (RTM) or other suitable material.

21. A material according to Claim 1 prepared in the place of eventual use by deposition of successive components on a machine part or like object.

22. A method of preparing a material as claimed in Claim 1 including overlaying the second component on the first, providing a well-defined boundary where the second component is contiguous with the first, and causing or producing a stress-free strain in the overlaid component, at least adjacent the first component.

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23. A method of preparing a material as claimed in Claim 1 including overlaying the second component on the first, ppermitting or causing the second component structure to be influenced by the first to produce said cooperation.

24. A magnetic material of a first component as a layer and a second component as a layer overlaid on the first, there being a lattice structure in at least one of said layers and in which magnetic material said lattice structure is modified where said first and second component layers are contiguous whereby the magnetostrictive property of the material is enhanced.

25. A material according to Claim 24 in which there is an interface region where the layers are contiguous.

26. A material according to Claim 25 in which the modification is in the interface region.

27. A material according to Claim 25 in which the interface region is in an elemental layer and the lattice structure in at least said region is one other than the usual one for the later element.

28. A material according to Claim 24 in which a third component as a layer is overlaid on the second and a lattice structure of said third layer is modified in its turn.

29. A material according to Claim 24 including further components as layers overlaid with or without lattice structure modification.

30. A material according to Claim 24 in which a said layer has a lattice structure modified by an overlaid layer, at least contiguous to the overlaid layer.

31. A material according to Claim 24 in which each of the component layers has an inherent lattice structure and said modification is that at least one inherent structure is modified by another contiguous inherent structure.

32. A material according to Claim 31 in which the modification is that one lattice, at least where contiguous to another lattice, adopts at least approximately the another lattice form.

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33. A material according to Claim 24 including a first layer and the overlaid second layer identified as a bilayer.

34. A material according to Claim 33 having at least thirty bilayers.

35. A material according to Claim 34 in which each layer of a bilayer is at least a few nanometres thick, that is a few tens of atoms thick, and up to ten or more nanometres thick.

36. A method of producing a multilayer magnetic material including depositing a first component, causing or permitting a second component to be progressively overlaid on the first, controlling the depositing of the first component and the overlaying of the second component to produce respective different structures and causing or permitting the structures to cooperate to assist the magnetostrictive behaviour of the material.

37. A method of producing a multilayer magnetic material including providing a first component layer of a first lattice structure, causing or permitting a second layer of material of a second lattice structure to be progressively overlaid on the first material, controlling the overlaying of the second material so that at least one component layer in a region contiguous to the interface of the layers has a lattice structure modified by the overlaying action.

38. A method according to Claim 36 or Claim 37 including overlaying by sputtering.

39. A method according to Claim 36 or Claim 37 including overlaying substantially atom-by-atom.

40. A material according to Claim 1 or Claim 34 in which each component or layer of a bilayer is at least one nanometre thick and up to ten nanometres thick, that is a few atoms to a few tens of atoms thick.

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41. A multilayer material as claimed in Claim 1 and having an M/H loop characteristic which is altered by distorting the material, for example by bending.

42. A multiple multilayer magnetic material as claimed in Claim 1 including an insulating layer, to enhance the frequency response.

43. A magnetic material arrangement including a magnetic material as claimed in Claim 1 or Claim 24 or as produced in accordance with Claim 36 and means to cooperate with the material to convey to an external connection a response of the material to an external action.

44. An arrangement as claimed in Claim 43 in which the response is a change of permeability or dimension and the external action may be the stressing of the material.

45. An arrangement as claimed in Claim 43 in which the action is flexure of a support for the material or an ambient pressure change, or an external magnetic field.

46. An arrangement as claimed in Claim 44 forming part of an accelerometer, a torque sensor or like device.

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ABSTRACT

A magnetic material of at least two component parts arranged to have respective structures which mutually are not homogenous, the structure of one part cooperating with the structure of the other to assist [Fig. 3] the magnetostrictive behaviour of the material. The cooperation can be the structure of one part altering the atomic spacing of the other part [Fig. 1, Fig. 2]. The alteration can be an increase from the usual atomic spacing. There can be contiguous component parts and the cooperation can be a pseudo free surface on at least one said part or a reduced restriction of the local moment otherwise achieved by the contiguous component parts [Fig. 5]. A component can be at least partly an alloy and the alloy can have ratio other than the usual one for said alloy. One of the components can be non-magnetic.

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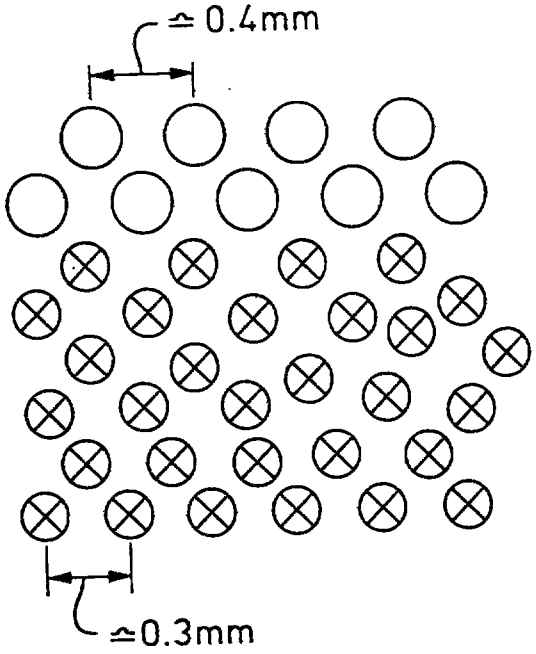


Fig. 1

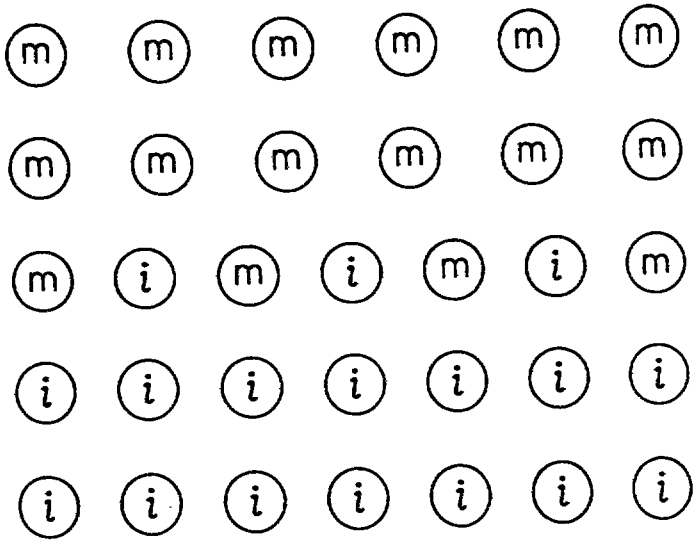


Fig. 2

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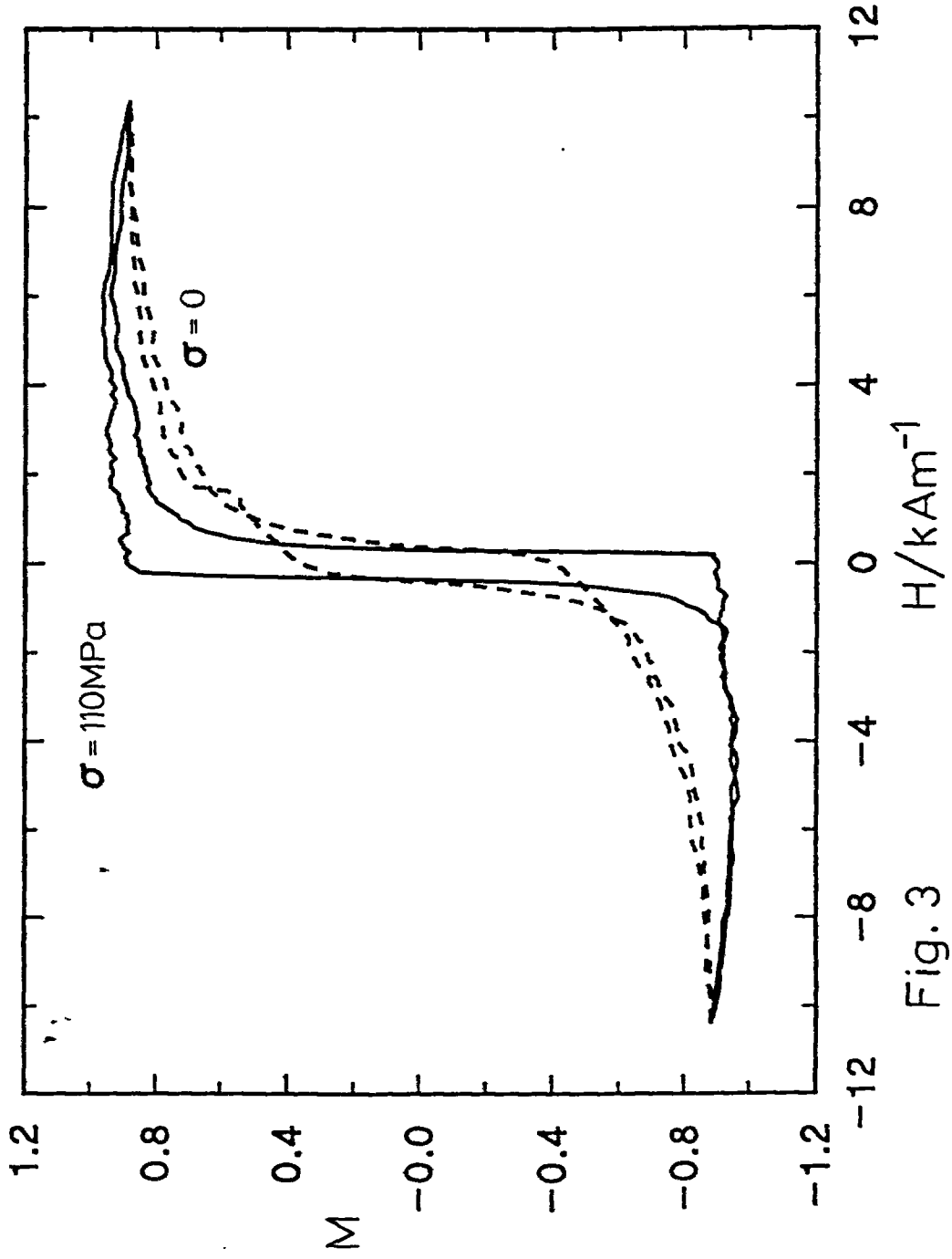


Fig. 3

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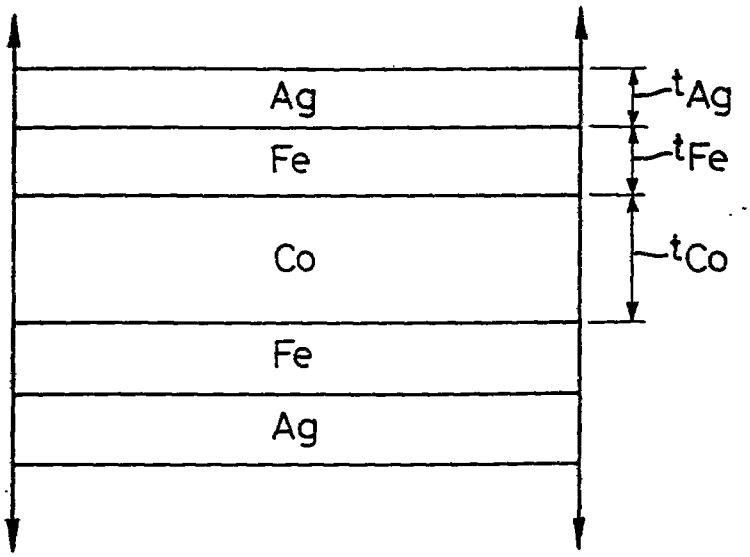


Fig. 4

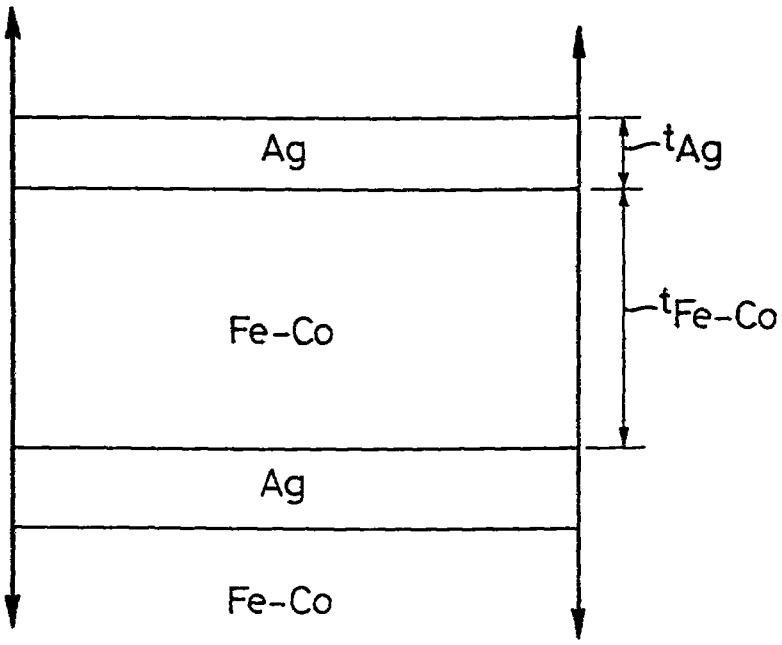


Fig. 5

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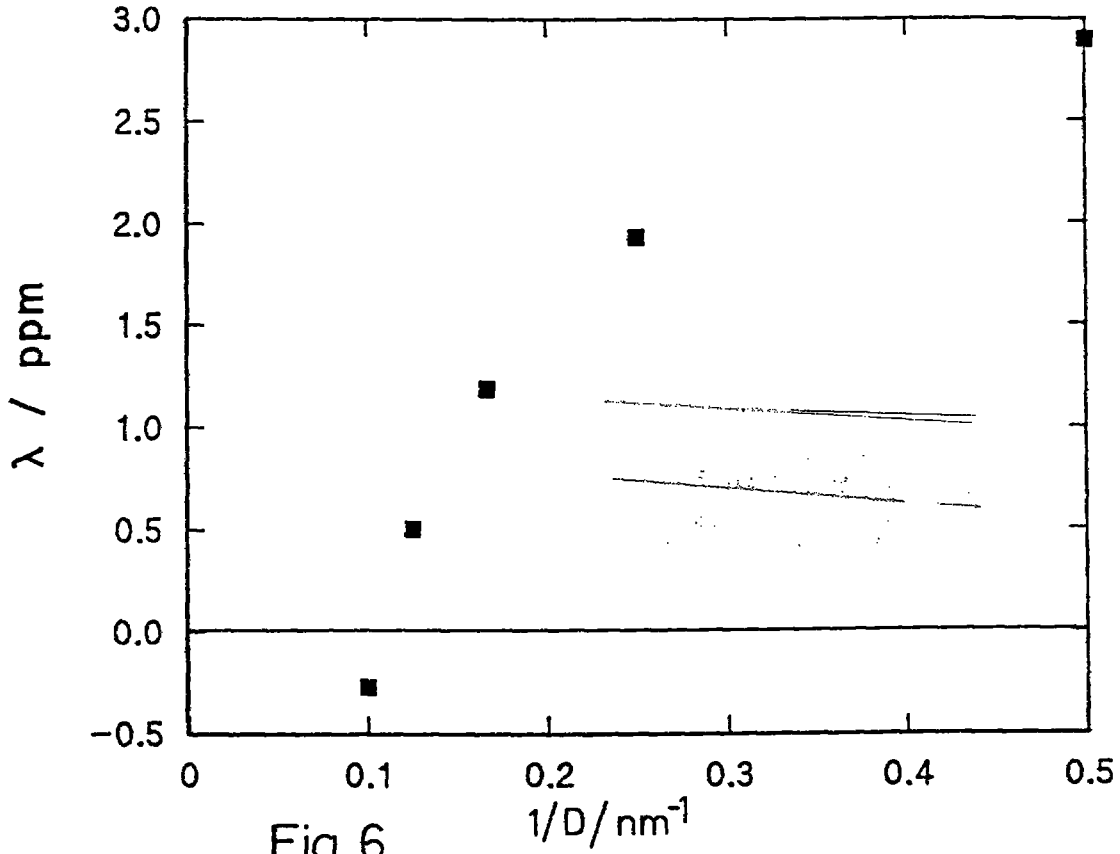


Fig.6

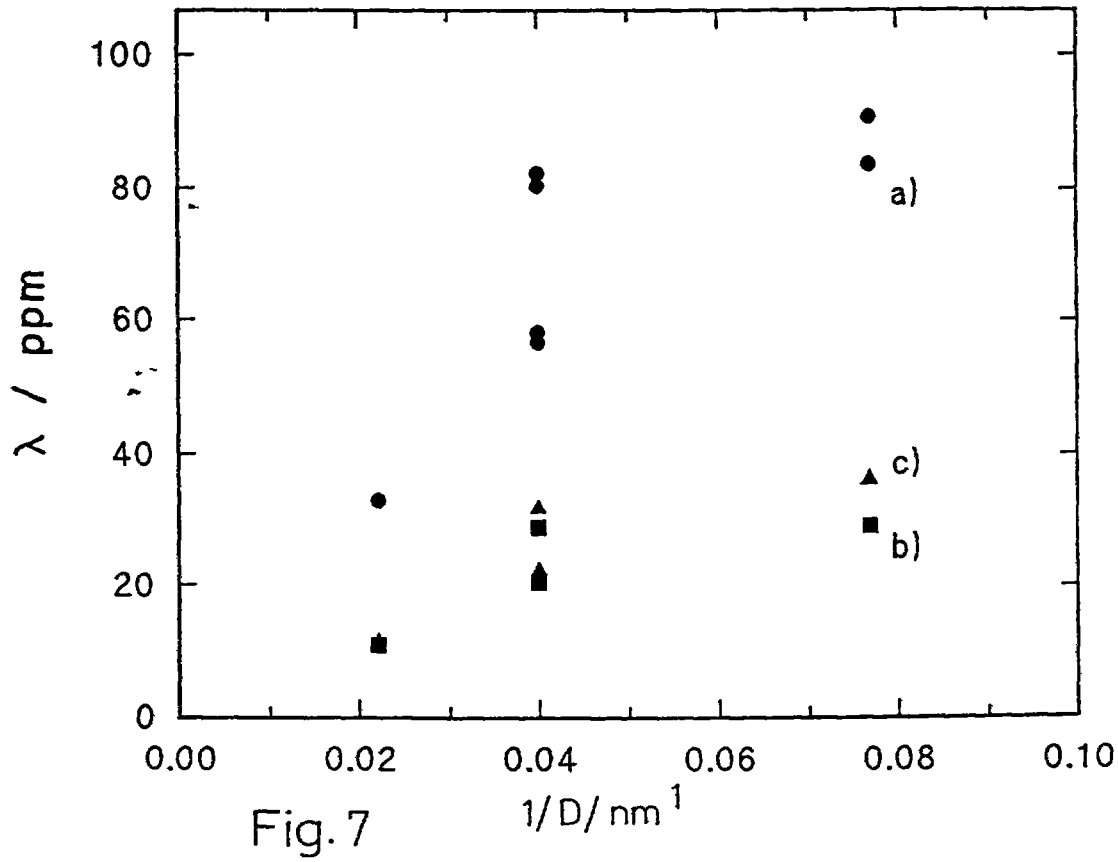


Fig. 7

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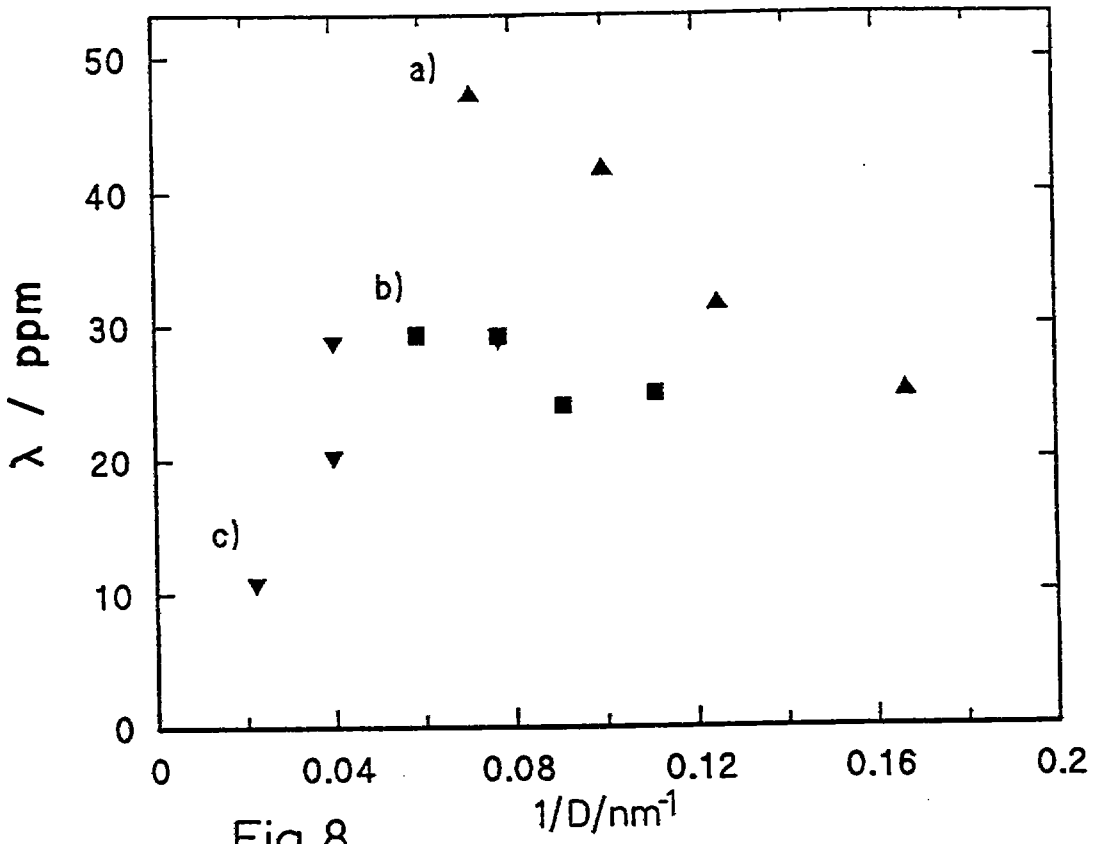


Fig.8

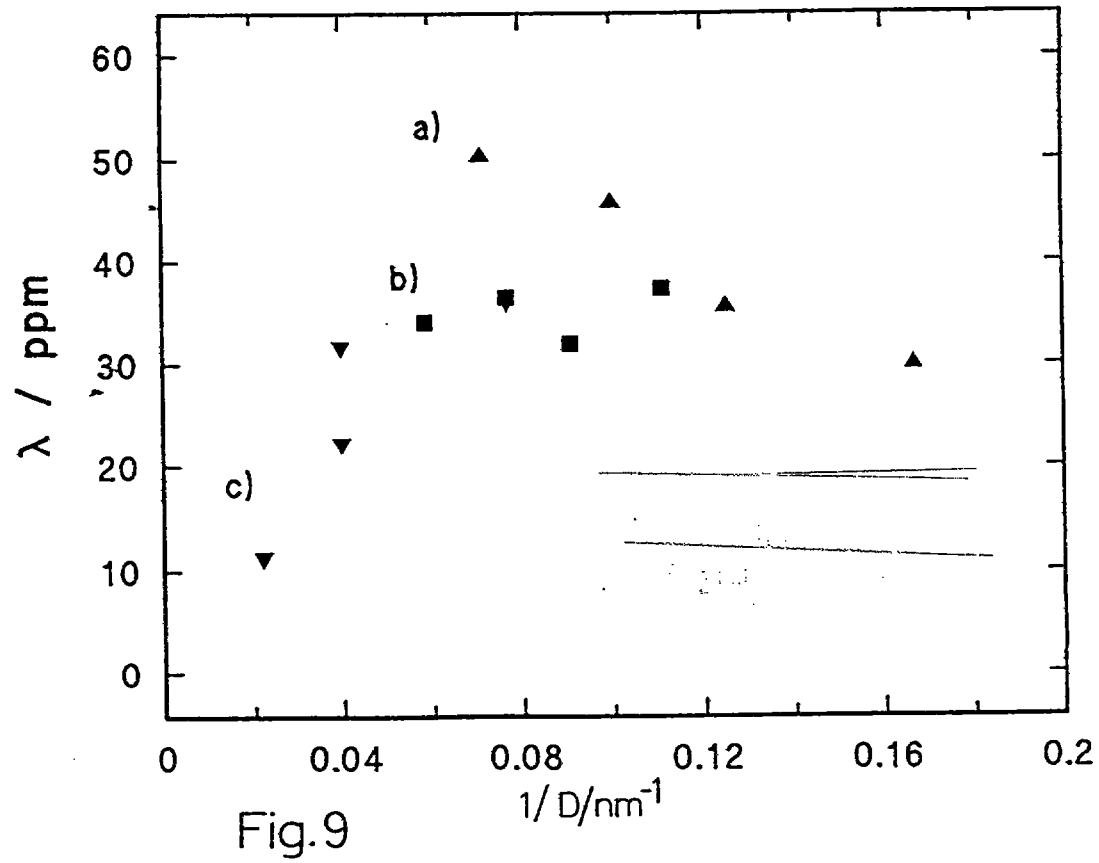


Fig.9

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	t(Fe)	t(Co)	t(Ag)		T_a	t_a
	(nm)	(nm)	(nm)			
S1	n		n	n = 1,2,3,4,5		
S2	n	2n	5	n = 1,2,5,10	290°C	30mins
S3	n	2n	5	n = 1,1.5,2,3	300°C	10-50mins
S4	n	2n	2	n = 1,1.5,2,3	300°C	10-50mins

Fig. 10

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MAGNETOSTRICTIVE MATERIAL

This invention relates to the production of materials with useful magnetostrictive properties.

Magnetostriction is the property which relates magnetic characteristics of a body of material to a change of the shape of the material. The property is seen in the change in size of bodies of certain materials when the magnetic environment changes or the change in magnetic characteristic when a force is applied to such a body to change its shape. Magnetostriction is a dimensionless quantity represented by the magnetostriction constant λ_s, relating magnetisation and shape change and in the SI system of units useful values are some tens or hundreds of parts per million, particularly for use in sensors and transducers. For such uses a high magnetomechanical coupling factor is desirable. Also "soft" magnetic properties are generally preferred.

While thin film amorphous alloys and magnetic multilayers individually provide some of the required properties there is still a strong need for a significant improvement in the properties available and for materials which exhibit a useful combination of such properties.

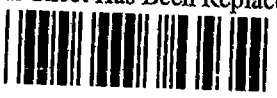
It is an object of the invention to provide materials with improved properties and methods of production of such materials.

According to the invention there is provided a magnetic material of at least two component parts arranged to have respective structures which mutually are not homogenous, the structure of one part cooperating with the structure of the other to assist the magnetostrictive behaviour of the material.

The cooperation may be a pseudo free surface on a part.

The cooperation may reduce restriction of the local moment of a part.

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The cooperation may be the structure of one part altering the atomic spacing of the other. Preferably the alteration is an increase from the usual spacing. The structure may be the absence of a regular lattice.

There may be an interface region in one component where said first and second components are contiguous. There may be an interface region where the layers are contiguous. The modification may be in the interface region. Where the interface region is in an elemental component the lattice structure may be one other than the usual one for the elemental component. Where the interface region is in a component which is at least partly an alloy the alloy may have a ratio other than the usual one for said alloy.

A third component layer may be overlaid on the second and a lattice structure of said third layer may be modified in its turn. Further component layers may be overlaid with or without lattice structure modification.

A layer may have a lattice structure modified by an overlaid layer, at least contiguous to the overlaid layer.

Each of the component layers may have an inherent lattice structure and modification may be that at least one inherent structure is modified by another contiguous inherent structure.

The modification may be that the second lattice, at least where contiguous to the first component layer, adopts at least approximately the first lattice form.

One of the first and second component layers may be a magnetic component and the other may be magnetic or a non-magnetic component. While each of the first and second layers will generally be different elements or alloys it is also possible magnetic and non-magnetic forms of one component may be used.

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The magnetic component may be chosen from the group of ferromagnetic elements of at least iron, nickel, cobalt, gadolinium and terbium.

The non-magnetic component may be chosen from aluminium, magnesium, gold and silver and silicon oxide.

Preferably there are many first and second layers in the material forming a multilayer material. Generally there will be alternating said first and second layers. One first layer and the overlaid second layer may be identified as a bilayer. A first and second layer may have a third layer associated therewith as a trilayer and may further have a fourth layer associated therewith as a quadlayer. In general the material form can be expressed as of M multilayers each having N component layers. Typically N will be between two and five while M will have a value of a few tens, typically 20 to 50.

There may be a substrate on which the multilayer material is built up. The material may remain on the substrate for use or be released therefrom, for example by chemical means. The substrate may be glass or mica or KAPTON (RTM) or other suitable material. The magnetic material may be prepared in the place of eventual use, for example by deposition of successive component layers on a machine part or like object. There may be more than 30 bilayers.

Each layer of a bilayer may be at least a few nanometres thick, that is a few tens of atoms thick, and may be up to ten or more nanometres thick. It is believed that a well-defined boundary is useful where the second layer is overlaid on the first. This will produce a stress-free strain in the overlaid layer.

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One form of the material described above may prepared by a method including overlaying the second component on the first, providing a well-defined boundary where the second component is contiguous with the first, and causing or producing a stress-free strain in the overlaid component, at least adjacent the first component.

Another form of the material described above may prepared by a method including overlaying the second component on the first, permitting or causing the second component structure to be influenced by the first to produce said cooperation.

According to a particular aspect of the invention there is provided a magnetic material of a first component layer and a second component layer overlaid on the first, there being a lattice structure in at least one of said layers and in which magnetic material at least one lattice structure is modified where said first and second component layers are contiguous whereby the magnetostrictive property of the material is enhanced.

There may be an interface region where the layers are contiguous. The modification may be in the interface region. Where the interface region is in an elemental layer the lattice structure in at least said region may be one other than the usual one for the layer element.

According to the invention there is provided a method of producing a multilayer magnetic material including depositing a first component, causing or permitting a second component to be progressively overlaid on the first, controlling the depositing of the first component and the overlaying of the second component to produce respective different structures and causing or permitting the structures to cooperate to assist the magnetostrictive behaviour of the material.

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The method may include providing a first component layer of a first lattice structure, causing or permitting a second component layer of a second lattice structure to be progessively overlaid on the first, controlling the overlaying of the second component so that at least one component layer in a region contiguous to the interface of the layers has a lattice structure modified by the overlaying action.

The method may include overlaying by sputtering. The method may include overlaying substantially atom-by-atom. The method may include sputtering from a target which may be of one element or of two or more elements to enable the deposit of an alloy.

The method may include causing or permitting the respective different structures to be distinct and form an interface which provides a pseudo free surface.

A multilayer material as described above has an M/H loop characteristic which is altered by distorting the material, for example by bending.

According to a particular aspect of the invention there is provided a multiple multilayer magnetic material as described above in which the overlaid second layer is covered by an insulating layer, to enhance the frequency response.

According to the invention there is provided a magnetic material arrangement including a magnetic material as described above and means to cooperate with the material to convey to an external connection a response of the material to an external action.

The response may be a change of permeability or dimension and the external action may be the stressing of the material. The action may be flexure of a support for the material or an ambient pressure change, or an external magnetic field.

The arrangement may form part of an accelerometer, a torque sensor or like device.

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Embodiments of the invention will now be described with reference to the following examples and drawings, in which:

Figure 1 shows in schematic form a representation of a lattice structure of a multilayer material according to the invention,

Figure 2 shows in schematic form a representation of another lattice structure of a multilayer material according to the invention,

Figure 3 shows M/H characteristics of a material according to the invention,

Figures 4 and 5 show in schematic form a portion of a multilayer material, after deposition and after subsequent annealing respectively,

Figures 6 to 9 show plots of characteristics of materials embodying the invention,

Figure 6 shows magnetostriction constant of Fe/Ag multilayers (S1) versus 1/(modulation period),

Figure 7 shows magnetostriction constant of Fe-Co/Ag multilayers versus 1/(modulation period) calculated using E_f from both the volume average and the vibrating reed correction from equation 5, (squares and circles respectively). Also shown are the effective magnetostriction constants of the alloy layers alone, using the volume average E_f method (triangles),

Figure 8 shows magnetostriction constant of Fe-Co/Ag multilayers versus 1/(modulation period),

a) t_{Ag} = 2 nm (S4),

b) t_{Ag} = 5 nm (S3),

c) t_{Ag} = 5 nm, t_{anneal} = 30 minutes (S2),

Figure 9 shows magnetostriction constant of Fe-Co layers within Fe-Co/Ag multilayers versus 1/(modulation period), (a), (b) and (c) as in Figure 8, and

Figure 10 is a table of materials exemplifying the invention.

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Various proposals for multilayer materials having magnetic properties have been made, for example de Wit, Witmer and Dirne (Advanced Materials, Vol. 3 (1991) No. 7/8 pp 356 to 360) and Zeper, van Kesteren and Carcia (Advanced Materials, Vol. 3 (1991) No. 7/8 pp 397-399). In the first of these (de Wit, etc) a material with enhanced saturation magnetisation and relative permeability but minimal magnetostriction, specifically for a video recorder read/write head, is described. To achieve this a very small grain size is sought for the magnetic material layer (iron, grain size below 10 nanometres) and to produce such a grain size the iron layers are around 10 nanometres thick. The layers are separated by thinner layers of another ferromagnetic material, specifically an iron/chromium/boron layer. This layer needs to be at least two nanometres thick to prevent the grains in one iron layer linking with those in another iron layer by columnar growth and specifically epitaxial growth is not desired. In the second (Zeper et al) a magneto optical recording material is described, for enhanced recording density, which has adequate Kerr rotation and low enough Curie temperature while having a preferred magnetisation direction perpendicular to the material layer. This preferred direction only occurs with cobalt/platinum or cobalt/palladium multilayers when the cobalt layers are less than some 0.8 to 1.2 nanometres. The thickness of the non-magnetic but magnetically polarisable platinum or palladium layer is set by the required balance of Kerr effect and Curie temperature and for platinum is about 0.9 nanometres with a 0.4 nanometre cobalt layer. The cobalt layer is about two atom layers thick so that the required magnetic anisotropy is not reduced by "bulk" atoms between the surface layers. In Zuberek, Szymczak, Krishnan and Tessier (Journal de Physique, Vol. 12, No. 9, Colloque C8, December 1988, pp 1761 to 1762) it is suggested that a "bilayer" of evaporated component materials depends for

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effectiveness on the thinness of a nickel layer. In Dirne, Tolboom, de Wit and Witmer (J. Magn. Magn. Mat., No. 83, (1990) pp 399 to 400) the possibility of interface mixing in Fe/Co multilayers is discussed and seen as a disadvantage. Techniques to avoid this disadvantage are suggested.

Multilayers of the present invention do not follow the techniques of the above proposals. The present invention provides in one aspect that a layer of a first component overlaid on another layer, of a second component, is caused in a region contiguous with the other layer to adopt a structure other than that of the lattice which it would normally adopt. In this way, which can be described as lattice mismatch, a stress-free strain can be produced in at least the contiguous region of the overlaid layer. A result of this stress-free strain is an enhanced magnetostriction constant, λ_s.

Typically such layers of a first material overlaid on the second would be arranged as several bilayers on a substrate. Each layer of a bilayer would be some five to fifteen nanometres thick and deposited by sputtering so that atoms being deposited arrive at a rate slow enough to be able to be influenced in deposit position by the existing lattice of the second component layer or that of the first-deposited overlaid first component atoms. Care is needed to avoid building stress into the layers by the thermal effects of sputtering. Some ten to fifty bilayers can be deposited. The overlaid component applied adjacent to the second component lattice is conveniently caused to adopt a lattice spacing appropriate to that of the second component. This will cause a stress-free strain in at least the contiguous region of the overlaid component. The stress-free strain will be best exhibited if there is a sharp boundary between the layers.

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If required a pre-stress can be built in to the eventual material by, for example, keeping the substrate hot during deposition of components so that the substrate contracts on cooling. Also a magnetic bias can be built in by including a layer of permanently magnetisable component for example as or immediately above the substrate. A buffer substrate can also be used but care must be taken that the buffer transmits any strain that is applied for action by or on the magnetostrictive material. An inert or insulating layer may be applied to protect the material. Films may be grown under the influence of an imposed magnetic field to enhance the desired effect.

Figure 1 represents in a schematic manner the lattice at and near the surface of the second component layer and the lattice in the contiguous region of the overlaid component layer. In the Figure overlaid component atoms are indicated by the smaller, crossed, circles and the second layer component atoms by the more widely spaced larger, open, circles.

To investigate the magnetostrictive properties further examples have been prepared. Typically these are a number of alternate layers of magnesium and iron, each a few nanometres thick and having a few tens of bilayers. The layers are not necessarily of the same thickness. The layers are deposited by sputtering from alternately exposed sources using conventional techniques. The preparation is arranged to ensure that bulk effects are not significant. The effect of stress on the film on the shape of the magnetisation/field (M/H) loop was also examined. A small amount of distortion, by bending the specimens, changed the loop shape by a detectable amount, increasing the value of M at lower values of H compared with the loop for the unstressed specimens.

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Other structures may be used to achieve the invention. Certain combinations of overlaid component layers will produce a degree of alloying and this in turn can produce "dislocations" in an atomic level structure which would otherwise be adopted. Provided one component layer is magnetic the other can be an insulator as the merging of the layers where they are contiguous can alter the spacing of the magnetic component layer lattice and thus usefully alter the magnetostrictive property. Figure 2 shows in sketch form this action where atoms of a magnetic component, identified at m, are displaced by the presence of atoms of another component, which may be non-magnetic even an insulator, identifed at i.

The present invention provides in another aspect an interface effect which can be a relatively sharp lattice mismatch as the overlaid components cannot easily mix or in a yet further aspect a smoother mismatch which may be an intermetallic region as overlaid components merge. Component layers need not be deposited separately, the rate of a first layer may be reduced while the rate of a second layer is increased to take over.

An insulating layer, for example silicon oxide, can be introduced to produce an improvement in performance, especially an improved high frequency performance. The insulating layer thickness will be dependent on the frequency at which the device is to operate but will also be dependent on the effect of the relative thickness of the insulating layer and the bilayer on the propagation velocities in the insulating and magnetic components. From these considerations it is possible to derive suitable thickness relationships for a particular application of the multilayer material. In general the insulating layer thickness will be several times that of the individual magnetic component layer. In this way

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magnetostrictive materials with several tri- or quad- layers can be built up, of the general form of L multilayers each having N component layers. At least one of the N component layers will be a magnetic, generally a ferromagnetic, substance such as iron, nickel, cobalt, gadolinium or terbium while another will be a non-magnetic substance and may be a metal or an insulator, examples including aluminium, magnesium gold and silver and silicon oxide. If N is more than two one of the non-magnetic layers can be a metal and another an insulator. A further magnetic component layer may be included. A magneto-electric transducer can be formed by placing structures as exemplified above onto an electrostrictive structure such as PVDF, the dimensional changes in the magnetic structures imposing a strain on the electrostrictive structure, producing an electrical signal related to the action on the magneto strictive structure.

Further examples of the invention will now be described.

Multilayers are grown using an rf glow discharge sputtering system in a sputter-up configuration with three 15 cm water-cooled magnetron target electrodes on a carousel, and a water-cooled substrate platen electrode which allows the substrate to be pre-sputtered. The substrate platen also has a heater capable of temperatures up to 600°C. The target-substrate distance was set at 60 mm, with an argon plasma pressure of 5 mtorr. The sputtering equipment is a NORDIKO machine. The deposition rates were calibrated using a "Talystep" stylus based film thickness measurement instrument. Structural characterisation was also done using X-ray diffraction.

The substrates were glass and Kapton (Registered Trade Mark) (polyimide). The substrate for SAMR measurements (see below) was 25 mm thick Kapton, which was thin enough and had a low enough Young's modulus to avoid overly constraining the films while still being able to support them.

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Fe/Ag multilayers were grown with 50 repeats (i.e. 50 bilayers).

Fe-Co/Ag multilayers of 25 repeats (25 bilayers) were grown. The layers of Fe-Co alloy in the Fe-Co/Ag multilayers were produced by growing sandwiches of Fe/Co/Fe between the silver layers (Figure 4). The multilayer was subsequently annealed to allow interdiffusion of the Fe and Co to form a layer of alloy of thickness t(Co)+2t(Fe) (the Ag is immiscible with both) (Figure 5). The heat treatment was done in a non-inductively wound tube furnace, with the sample laid flat between two glass slides in a vacuum provided by a rotatry pump and nitrogen trap to eliminate oxidation. The anneal time and temperature were 30 minutes and 290°C respectively.

A series (S1) of five Fe/Ag multilayers was grown where t(Fe)=t(Ag)=1,2,3,4,5 nm.

A series (S2) of Fe/Co/Fe/Ag (i.e. Fe-Co/Ag) multilayers was grown where the thickness of the Co layer was twice that of each of the Fe layers so that the total thickness was the same for both, resulting in a post-anneal alloy of a nominal composition of $Fe_{50}Co_{50}$. The thickness of this layer was varied from 4 to 40 nm and t(Ag) was set at 5 nm. These were subsequently annealed in vacuum for 30 minutes at 290°C. Two other Fe/Co/Fe/Ag (i.e. Fe-Co/Ag) series were grown with silver layer thicknesses of 2 (S4) and 5 (S3) nm. The anneal times were longer for the films with thicker Fe/Co layers because of the greater degree of interdiffusion required. The anneal times (at 300°C) were 10, 20, 30 and 50 minutes for alloy layer thicknesses 4, 6, 8, 12 nm respectively.

As the layer thicknesses are decreased the volume fraction of the interface regions is increased. This increases the proportion of the multilayer material which is formed by atoms at or near the surface of a layer. The local moment is increased in the vicinity of a free surface so the local moment effect is enhanced as the interface volume fraction is increased. The texture of a layer is also significant and its

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retention can be enhanced by depositing the alloy directly and avoiding or reducing the annealing step. This will also encourage growth of the alloy in the [110] direction normal to the layer.

The coercive fields of Fe-Co/Ag multilayers are between 1 and 2 kAm^{-1}, reducing with layer thickness. Generally, the annealing rendered the multilayers magnetically softer. Also by annealing the multilayers were stress-relieved and the iron and cobalt layers were caused to mix more thoroughly. The interfaces with silver were probably sharpened, since silver is immiscible with iron and with cobalt. For the thinnest iron and cobalt layers (D=9nm, i.e. t_{Co}=2nm) the region between silver layers would have been almost completely alloyed as-deposited [Dirne and Denissen, 1989]. Annealing this multilayer increased (approximately doubling) its coercive field in distinction to the other multilayers, and it became impossible to measure the magnetostriction. This is considered below.

The length of an anneal required to achieve complete alloying at a given temperature will depend on the thickness of the component layers. Thus it is possible that the anneals did not completely alloy the layers, especially in the thicker multilayers, and that they were longer than required for the thinner multilayers. The optimum heat treatment may not have been used.

Magnetostriction measurements were made using the Small Angle Magnetisation Rotation method proposed by Norita et al. [1980] and widely used on amorphous ribbons.

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The basic principle of the method is as follows: the sample to be measured is saturated in the longitudinal direction (z) by a large dc field. A smaller ac field of frequency ω acts in the transverse direction (y). This results in a small degree of rotation of the macroscopic moment direction about the longitudinal direction. If a stress, σ, is also applied to the sample along this direction, the angle of moment rotation is given by

$$\theta = \frac{\mu_o M_s H_y}{H_z + H_d + H_\sigma} \qquad (1)$$

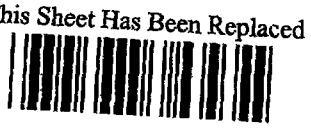
where H_d is the demagnetising field due to the sample shape, and H_σ is the stress-induced anisotropy field. This is given by

$$H_\sigma = \frac{3\lambda_s \sigma}{\mu_o M_s} \qquad (2)$$

The sample is mounted within a search coil whose axis is in the longitudinal direction. Thus a rotation of the magnetisation about this axis results in an induced ac voltage across the search coil with frequency 2ω, $V_{2\omega}$.

A change in the stress on a magnetostrictive material effects a change in the stress induced anisotropy field. In the case of a positively magnetostrictive material, a tensile stress tends to make the stress axis magnetically easier, so aligning the moments towards this axis. This would cause the angle of moment rotation to decrease, so decreasing the search coil output voltage.

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This change in output voltage can be compensated by adjustment of the dc field, H_z. The change in H_z is then equal to the change in H_σ. Thus, the magnetostriction constant, λ_s, is generally determined by plotting the change in H_z for a series of applied stresses, so that (where moment rotation is small)

$$\lambda_s = \frac{\mu_o M_s}{3} \frac{dH_z}{d\sigma_a} \Big| V_{2\omega}, H_y \ . \qquad (3)$$

The multilayers are too thin to be self supporting and need to be measured while still on their substrates. Although these can have elastic moduli far lower than the multilayers, they are much thicker and so accommodate a significant proportion of the stress, so it is necessary to correct for their presence.

This correction can be made by assuming that the multilayer is strongly adhered to the substrate so that they strain by the same amount and that the Young's modulus, E, of the whole is equal to the volume average of the component layers and the substrate. This assumes that the reed as a whole (i.e. substrate + multilayer) is effectively homogeneous. In this case the magnetostriction is given by

$$\lambda_s = \frac{\mu_o M_s}{3} \left[\frac{E_{av} A_{f+s}}{E_f} \right] \frac{dH_z}{d\sigma_a} \Big| V_{2\omega}, H_y \ . \qquad (4)$$

The same method of correction was also used by Wenda et al. [1987] on measurements of FeB and FeSiB single films sputtered onto polyester substrates.

Measurements of multilayer samples were taken on two slightly different SAMR apparatus arrangements. The dc field was applied using a solenoid, while the ac field was applied with a pair of Helmholtz coils. The sample was placed within a search coil (4000 turns) coaxially with the solenoid, in the centre of the Helmholtz coils. In the second configuration, a second, "bucking", coil was also present and was backed off

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against the search coil to cancel out any induced voltages common to both, i.e. due to sources other than the sample magnetisation.

The sample was clamped at both ends. The clamp at one end was fixed while that at the opposite end was connected to a cable which was loaded with known weights. The dc field was determined from the product of the current and the number of turns per unit length in the solenoid.

The values of B_s were obtained from literature, namely 2.0T for iron and 2.3T for $Fe_{50}Co_{50}$ alloy. The value of B_s for the whole multilayer was simply B_s for the relevant magnetic layer multiplied by its volume fraction.

The Young's modulus of the multilayers was measured using the vibrating reed method, which is fully described in Berry and Pritchet [1975]. It is based on the measurement of the resonant frequencies of various modes of cantilever vibration, f_n, of reeds clamped at one end.

Reeds were cut from the multilayer using a sharp blade and had free lengths and widths of approximately 13-20 mm and 1 mm respectively.

The value of E for the multilayer can be obtained from the value for the reed from the expression (from Berry and Pritchet):

$$\frac{f^2_{reed}}{f^2_{substrate}} = 1 + \frac{t}{d}\left[\frac{3E_f}{E_s} - \frac{\rho_f}{\rho_s}\right], \qquad (5)$$

where the subscripts f and s refer to the multilayer and substrate respectively, and t and d are the multilayer and reed thicknesses (t«d).

The value of λ_s for the multilayer was calculated from equation 4. In the case of the Fe-Co/Ag multilayers (t(Co)=2t(Fe)), two data sets are presented, corresponding to the two methods of calculating E_f. For the Fe/Ag data, only the values of λ_s derived from the assumption of a volume average E_f are shown.

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The data for λ_s is shown versus the reciprocal of the modulation period, D, in Figure 6. At 1/D=0 the value of λ_s should be that for bulk iron. The bulk value of λ_s of polycrystalline iron is -7 ppm, although this becomes more positive as it becomes more textured, so that perfectly textured polycrystalline iron with the [110] direction normal to the film surface has a value of λ_s of approximately 14 ppm. In practice, the measured value of λ_s for a single iron film will generally lie between these two values. As a check a thick (0.8 μm) iron film was deposited onto each side of a Kapton susbtrate (1.6 μm total). λ_s was measured to be -3.0 ppm.

As D decreases so that the volume fraction of the Fe/Ag interface layers increases, it is found that λ_s becomes more positive. Hence the presence of silver is seen to enhance the value of λ_s in iron, as expected.

A series of multilayers was grown with layers of Fe and Co of equal thickness (5 nm) with silver layers of varying thickness. Because there was approximately twice the amount of iron as cobalt in the alloy layer after annealing, it is probable that there was a composition gradient across the layer, with a corresponding variation in λ_s. The λ_s data from this series of multilayers did not show any systematic trend and there was a wide scatter in the data and is therefore not shown here.

The λ_s data for the second series of multilayers is shown for these films versus the reciprocal of the modulation period in Figure 7. Also shown is the effective λ_s of the magnetostrictive material alone, derived via the data assuming a volume average Young's modulus. There are pairs of data points obtained via the Berry and Pritchet E_f correction method for most samples, corresponding to two values of E_f obtained from the third and fourth modes of reed vibration. There are two samples measured with t(alloy)=30 nm since two sections

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were taken from the same substrate and were annealed independently. However, E_{reed} for this sample was only measured using a reed from one of the anneals, so the pair of points from this sample with the higher λ_s values may be subject to large error. This figure illustrates the difference in the data resulting from the two methods of correction of the Young's modulus.

Before anneal, most samples were found to have small negative magnetostriction (generally smaller than -5 ppm), and large positive λ_s after anneal confirming that alloying of the component TM layers had alloyed. The sample with t(alloy)=4 nm gave a large positive λ_s before annealing, indicating that the iron and cobalt layers had intermixed to a high degree. It is suggested in Dirne and Denissen [1989] that Fe-Co alloys form at interfaces during deposition, with thicknesses of approximately 1 nm This means that approximately 50% of the Fe-Co in this sample was intermixed before anneal. Unfortunately, no SAMR response could be obtained from the sample after anneal. As stated earlier, this was accompanied by a remarkable increase in coercive field. It may be that since the layers were so thin, the anneal caused disruption of the coherency of the multilayer.

From the volume average data, it is seen that λ_s of the multilayer increases to approximately 20 ppm as the alloy thickness is reduced to 8 nm, equivalent to a value of λ_s of about 36 ppm for the alloy layers.

The magnetostriction of all three series (S2, S3, S4) of the Fe-Co/Ag multilayers were measured and are shown in Figure 8. These values were calculated using the volume average Young's moduli. The corresponding values of λ_s for the alloy alone are shown in Figure 9. A clear trend is seen for the series of multilayers (S4) with t_{Ag}=2 nm, where λ_s decreases with decreasing alloy layer thickness. The data for the series of multilayers with t_{Ag}=5 nm also show some

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reduction of λ_s with t_{Fe-Co}, but in Figure 8, the values of λ_s for the alloys are approximately constant (within error), indicating that the reduction in λ_s for the multilayer is due to loading by the silver layers. The reduction in λ_s for the t_{Ag} data set cannot be accounted for in this way.

The decrease of λ_s of the thicker multilayers from the series (S2) is thought to be due to the iron and cobalt layers not fully intermixing during the half-hour anneal. It should be noted that the values of λ_s for the two multilayers comprising layers of 2 nm Fe, 4 nm Co and 5 nm Ag, annealed at approximately 300°C for 30 minutes were measured to be the same within error. This adds confidence to the repeatability and accuracy of the data. If intermixing is poor composition gradients can arise.

The value of λ_s of the alloy layer was measured to be approximately 35±3 ppm. This compares with approximately 80 ppm for the bulk. A multilayer of Fe/Co was deposited without silver layers, and was annealed nominally to form a single layer (0.5 μm total thickness) of Fe-Co alloy, T_a=300°C, t_a=30 minutes. The value of λ_s of the film was measured to be 43 ppm. A further heat treatment of 30 minutes at 300°C was found to cause an increase in λ_s to approximately 65 ppm.

The magnetostriction constant obtained from SAMR values depends on the Young's modulus of the reed and also on that of the film only. Discrepancies have arisen between the values of E_{film} corrected from E_{reed} from two different methods of obtaining this correction. The method assuming a volume average of the moduli is thought to be the more reliable.

The values of λ_s of the Fe/Co/Fe/Ag multilayers were small and negative, as would be expected from an average of Fe and Co. After anneal, the λ_s values were large and positive, indicating that the Fe and Co had alloyed to a large extent. Annealing also had the effect of making the films softer, predominantly due to relief of internal stresses. Values of H_c of the post-anneal samples were typically of the order of 1kA/m.

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Figure 3 shows the effect of stress on the M/H loop for one of the multilayers from series S4. The specific one used is that where n = 3. The multilayer had 25 bilayers, each deposited as 3 nm Fe/6 nm Co/3 nm Fe/2 nm Ag, supported on a 25 micrometre Kapton layer. Annealing was 50 minutes at 300°C under vacuum. The dotted line is the M/H characteristic at stress $\sigma = 0$. When a stress of 110 MPa is applied the shape and size of the M/H loop changes very significantly, shown in the solid line. M is in arbitrary units.

In the case of the Fe/Ag multilayers, reduction in the thickness of the layers (i.e. an increase in the volume fraction of the interface regions) has the effect of making the magnetostriction more positive. The measured values of λ_s in this study increased from approximately -0.3 ppm for 5 nm Fe/5 nm Ag to approximately +3 ppm for 1 nm Fe/1 nm Ag. This is consistent with an increase in the local magnetic moment of iron at the Fe-Ag interfaces.

In the case of the Fe-Co multilayers, λ_s was seen to decrease with decreasing modulation period, where this was shown to be due to mechanical loading by the silver layers, where t_{Ag}=5 nm. When this correction was made, the value of λ_{Fe-Co} were found to be approximately 35 ppm, which is considerably lower than those of bulk Fe-Co alloys. Non-optimal heat treatments are thought to contribute to the reduction in the values, as indicated by the decrease in λ_s for the thicker multilayers annealed for 30 minutes. A definite decrease in λ_s with decreasing alloy layer thickness was seen for the multilayers where t_{Ag}=2 nm, which cannot be accounted for by the loading by the silver layers. The reason for this is not clear. It is possible that the silver layers are so thin that the alloy layers have percolated through, especially where these are appreciably thicker than the silver, so that the parameters of the multilayer approach those of a bulk material. It may be that inclusion of silver in the multilayer causes a reduction in λ_{Fe-Co}.

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It is seen from the above that thickness and crystallographic texturing of the alloy layer and silver layer loading all can affect the value of λ_s. Those skilled in the art will be able to proceed on the basis of the techniques set out above to produce multilayers with significant values of λ_s using appropriate component layers and dimensions.

Some of the above examples show that the technique of causing a magnetic component layer to adopt, in a surface region at least, a lattice structure other than the one it would normally adopt produces a magnetic material which exhibits a non-zero magnetostrictive coefficient, albeit at a coercivity more appropriate to a "hard" magnetic material than a "soft" one, but noting that this parameter was not controlled.

Others of the above examples show that the technique of causing a sharp lattice mismatch or smoother mismatch intermetallic region in overlaid component layers produces a magnetic material which exhibits a non-zero magnetostrictive coefficient. In particular a sharp lattice mismatch, such as occurs with immiscible component layers, could produce a pseudo free surface. The term pseudo free surface is used to describe the arrangement where the surface of a thin layer can emulate a free surface although another layer is contiguous.

The magnetic material can be used in sensing devices, such as pressure gauges, accelerometers and torque sensors, by attaching the material to a structure or by depositing the material in the place where it is to be used.

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CLAIMS

1. A magnetic material of at least two component parts arranged to have respective structures which mutually are not homogenous, the structure of one part cooperating with the structure of the other to assist the magnetostrictive behaviour of the material.

2. A material according to Claim 1 in which the cooperation is the structure of one part altering the atomic spacing of the other part.

3. A material according to Claim 2 in which the alteration is an increase from the usual atomic spacing.

4. A material according to Claim 1 in which the structure of at least one part is the absence of a regular lattice.

5. A material according to Claim 1 in which there are contiguous component parts and in which the cooperation is a pseudo free surface on at least one said part.

6. A material according to Claim 1 in which there are contiguous component parts and in which the cooperation reduces restriction of the local moment otherwise achieved by said contiguous component parts.

7. A material according to Claim 1 in which there is an interface region in one component where said first and second components are contiguous.

8. A material according to Claim 7 in which the interface region is in a component which is at least partly an alloy and the alloy has a ratio other than the usual one for said alloy.

9. A material according to Claim 1 in which one of the first and second components is a magnetic material and the other is a magnetic or a non-magnetic material.

10. A material according to Claim 1 in which the first and second components are different elements or alloys or are magnetic and non-magnetic forms of one element or alloy.

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11. A material according to Claim 10 in which a magnetic component is chosen from the group of ferromagnetic elements of at least iron, nickel, cobalt, gadolinium and terbium.

12. A material according to Claim 10 in which a component is chosen from the group of elements of at least aluminium, magnesium, silver and gold and silicon dioxide.

13. A material according to Claim 1 including many first and second components as layers.

14. A material according to Claim 22 including alternating said first and second components.

15. A material according to Claim 1 including a first and second component having a third component associated therewith as a trilayer.

16. A material according to Claim 15 having a fourth component associated therewith as a quadlayer.

17. A material according to Claim 1 having a material form expressed as M multilayers each having N components as layers, where M is between two and five while M has a value of a few tens, typically twenty to fifty.

18. A material according to Claim 1 including a substrate on which the material is built up.

19. A material according to Claim 18 in which the material remains on the substrate for use or is released therefrom, for example by chemical means.

20. A material according to Claim 18 in which the substrate is glass or mica or KAPTON (RTM) or other suitable material.

21. A material according to Claim 1 prepared in the place of eventual use by deposition of successive components on a machine part or like object.

22. A method of preparing a material as claimed in Claim 1 including overlaying the second component on the first, providing a well-defined boundary where the second component is contiguous with the first, and causing or producing a stress-free strain in the overlaid component, at least adjacent the first component.

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23. A method of preparing a material as claimed in Claim 1 including overlaying the second component on the first, ppermitting or causing the second component structure to be influenced by the first to produce said cooperation.

24. A magnetic material of a first component as a layer and a second component as a layer overlaid on the first, there being a lattice structure in at least one of said layers and in which magnetic material said lattice structure is modified where said first and second component layers are contiguous whereby the magnetostrictive property of the material is enhanced.

25. A material according to Claim 24 in which there is an interface region where the layers are contiguous.

26. A material according to Claim 25 in which the modification is in the interface region.

27. A material according to Claim 25 in which the interface region is in an elemental layer and the lattice structure in at least said region is one other than the usual one for the later element.

28. A material according to Claim 24 in which a third component as a layer is overlaid on the second and a lattice structure of said third layer is modified in its turn.

29. A material according to Claim 24 including further components as layers overlaid with or without lattice structure modification.

30. A material according to Claim 24 in which a said layer has a lattice structure modified by an overlaid layer, at least contiguous to the overlaid layer.

31. A material according to Claim 24 in which each of the component layers has an inherent lattice structure and said modification is that at least one inherent structure is modified by another contiguous inherent structure.

32. A material according to Claim 31 in which the modification is that one lattice, at least where contiguous to another lattice, adopts at least approximately the another lattice form.

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33. A material according to Claim 24 including a first layer and the overlaid second layer identified as a bilayer.

34. A material according to Claim 33 having at least thirty bilayers.

35. A material according to Claim 34 in which each layer of a bilayer is at least a few nanometres thick, that is a few tens of atoms thick, and up to ten or more nanometres thick.

36. A method of producing a multilayer magnetic material including depositing a first component, causing or permitting a second component to be progressively overlaid on the first, controlling the depositing of the first component and the overlaying of the second component to produce respective different structures and causing or permitting the structures to cooperate to assist the magnetostrictive behaviour of the material.

37. A method of producing a multilayer magnetic material including providing a first component layer of a first lattice structure, causing or permitting a second layer of material of a second lattice structure to be progressively overlaid on the first material, controlling the overlaying of the second material so that at least one component layer in a region contiguous to the interface of the layers has a lattice structure modified by the overlaying action.

38. A method according to Claim 36 or Claim 37 including overlaying by sputtering.

39. A method according to Claim 36 or Claim 37 including overlaying substantially atom-by-atom.

40. A material according to Claim 1 or Claim 34 in which each component or layer of a bilayer is at least one nanometre thick and up to ten nanometres thick, that is a few atoms to a few tens of atoms thick.

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41. A multilayer material as claimed in Claim 1 and having an M/H loop characteristic which is altered by distorting the material, for example by bending.

42. A multiple multilayer magnetic material as claimed in Claim 1 including an insulating layer, to enhance the frequency response.

43. A magnetic material arrangement including a magnetic material as claimed in Claim 1 or Claim 24 or as produced in accordance with Claim 36 and means to cooperate with the material to convey to an external connection a response of the material to an external action.

44. An arrangement as claimed in Claim 43 in which the response is a change of permeability or dimension and the external action may be the stressing of the material.

45. An arrangement as claimed in Claim 43 in which the action is flexure of a support for the material or an ambient pressure change, or an external magnetic field.

46. An arrangement as claimed in Claim 44 forming part of an accelerometer, a torque sensor or like device.

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MAGNETOSTRICTIVE MATERIAL

ABSTRACT

A magnetic material of at least two component parts arranged to have respective structures which mutually are not homogenous, the structure of one part cooperating with the structure of the other to assist (fig. 3) the magnetostrictive behaviour of the material. The cooperation can be the structure of one part altering the atomic spacing of the other part (fig. 1, fig. 2). The alteration can be an increase from the usual atomic spacing. There can be contiguous component parts and the cooperation can be a pseudo free surface on at least one said part or a reduced restriction of the local moment otherwise achieved by the contiguous parts (fig. 5'. A component can be at least partly an alloy can have ratio other than the usual one for said alloy. One of the components can be non-magnetic.

FIGURE 2.

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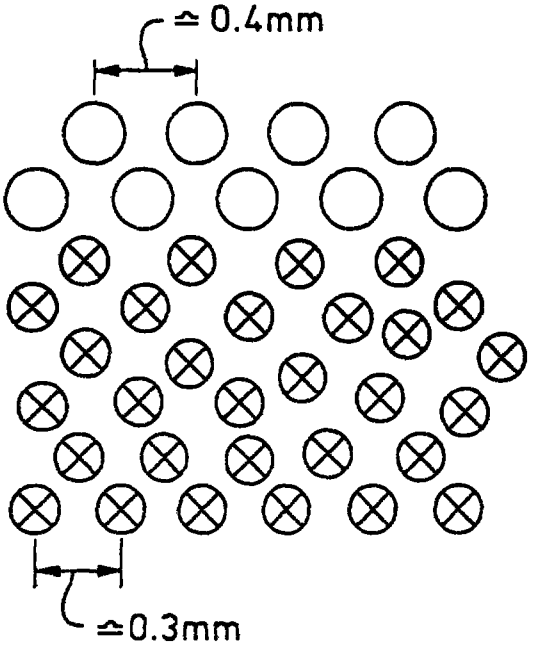


Fig. 1

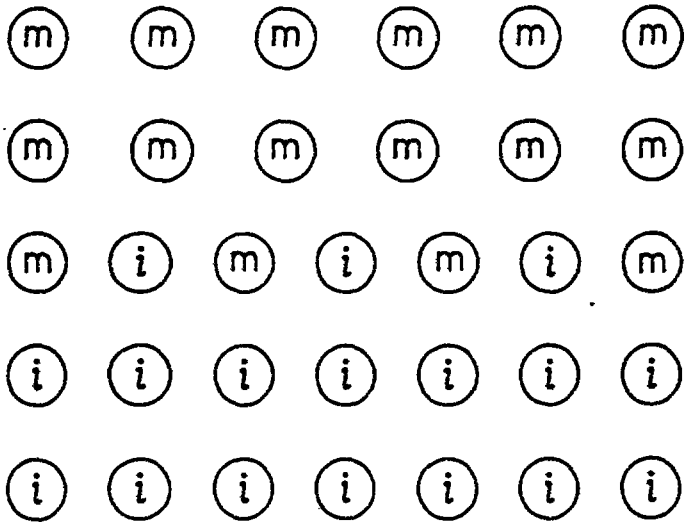


Fig. 2

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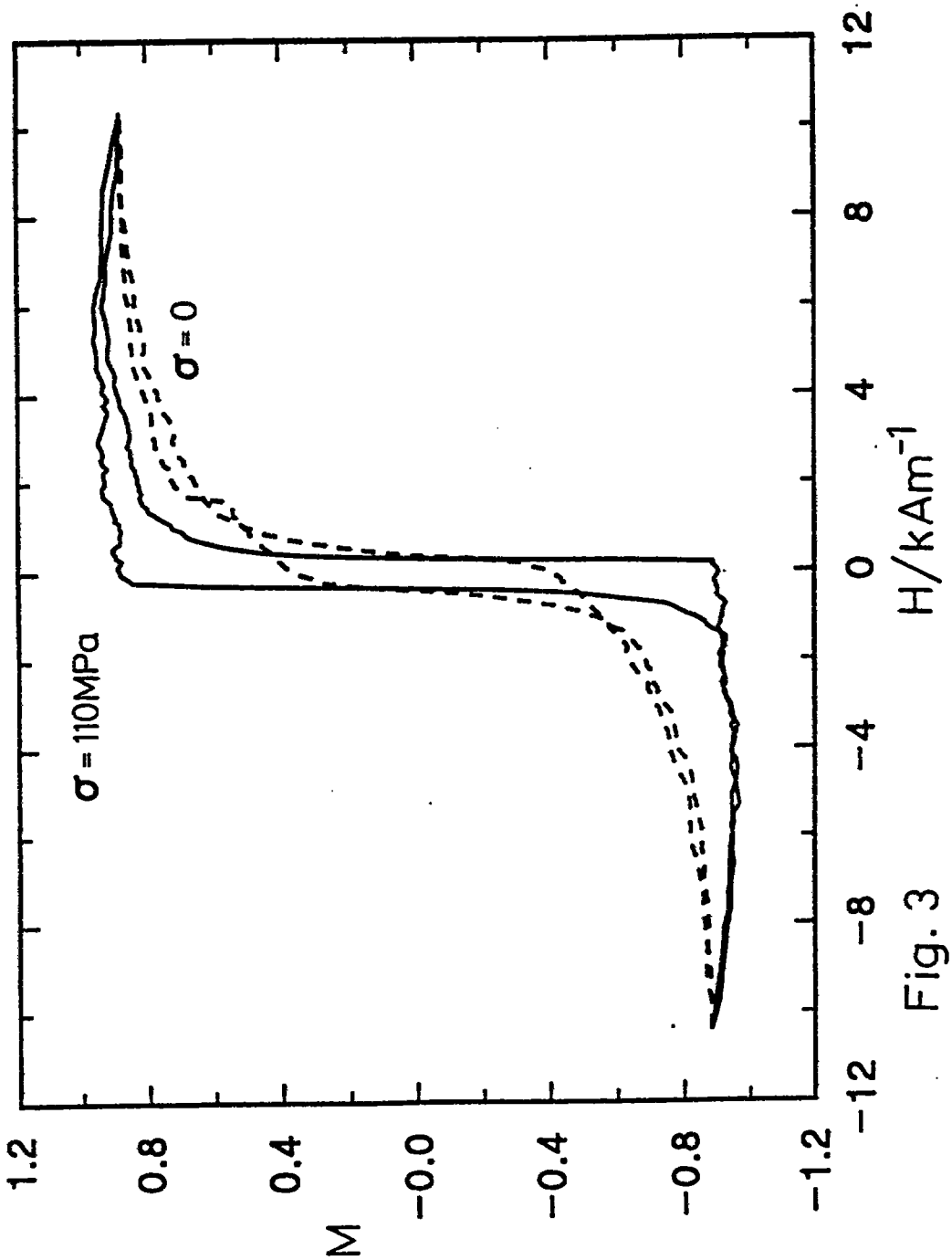


Fig. 3

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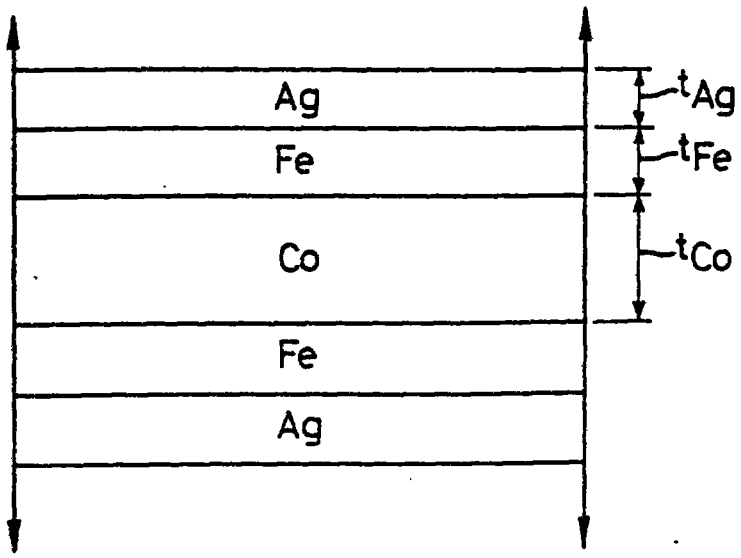


Fig. 4

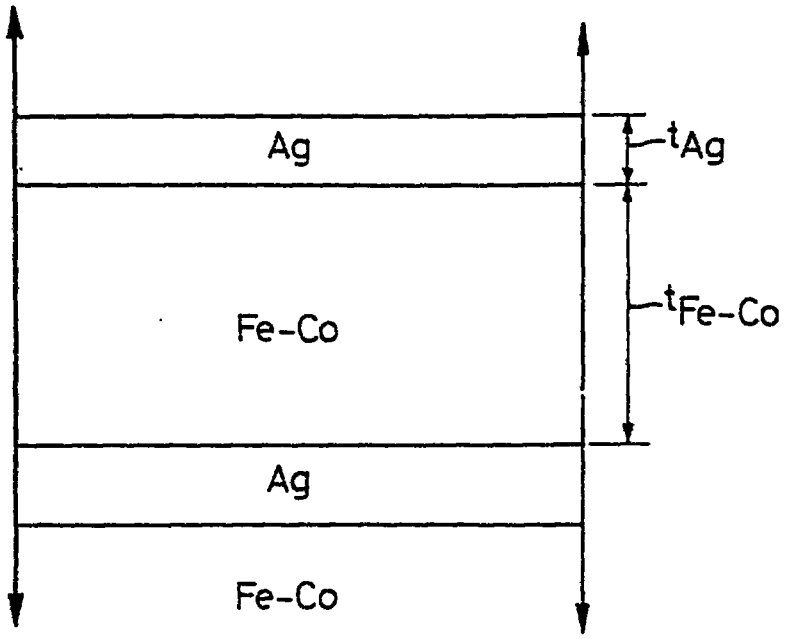


Fig. 5

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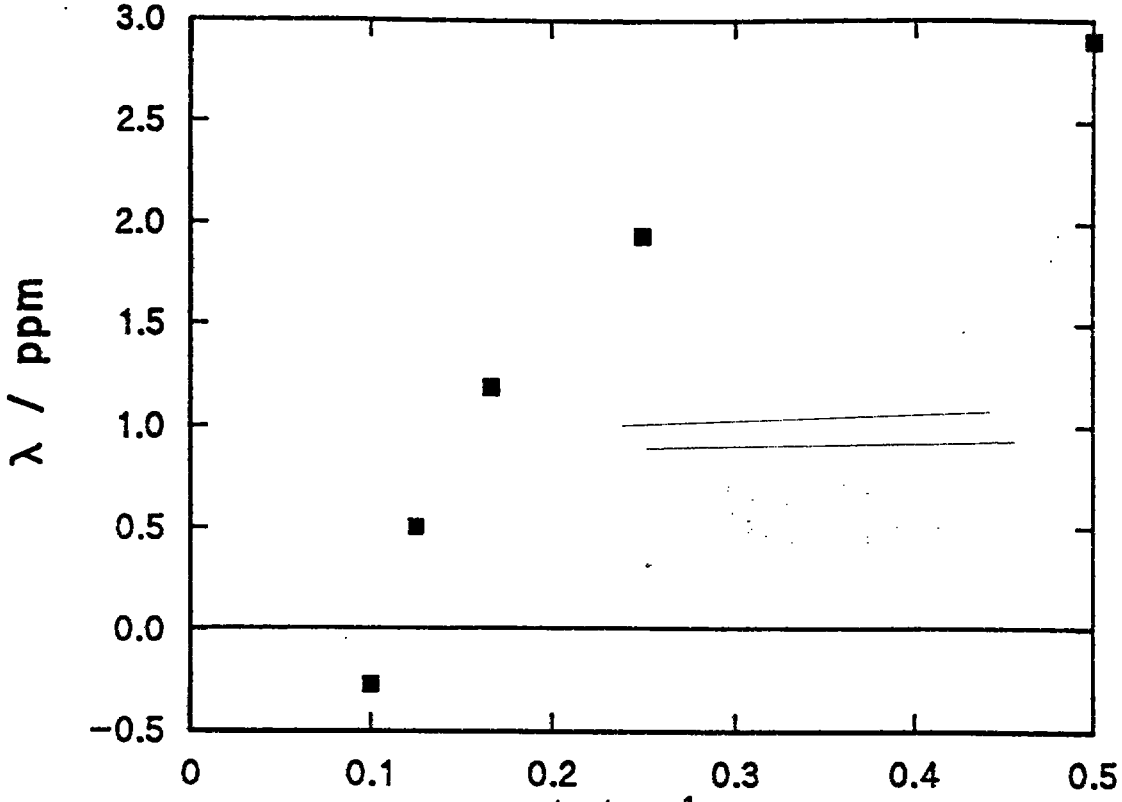


Fig. 6

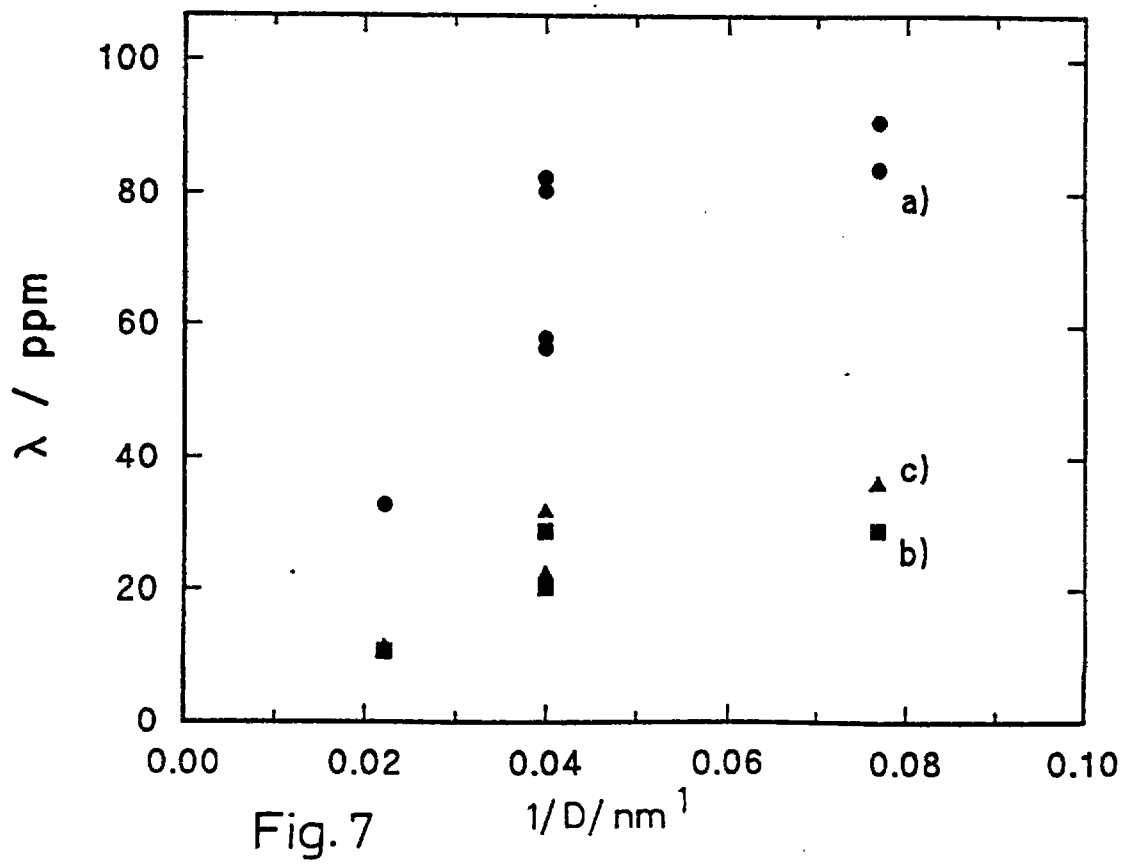


Fig. 7

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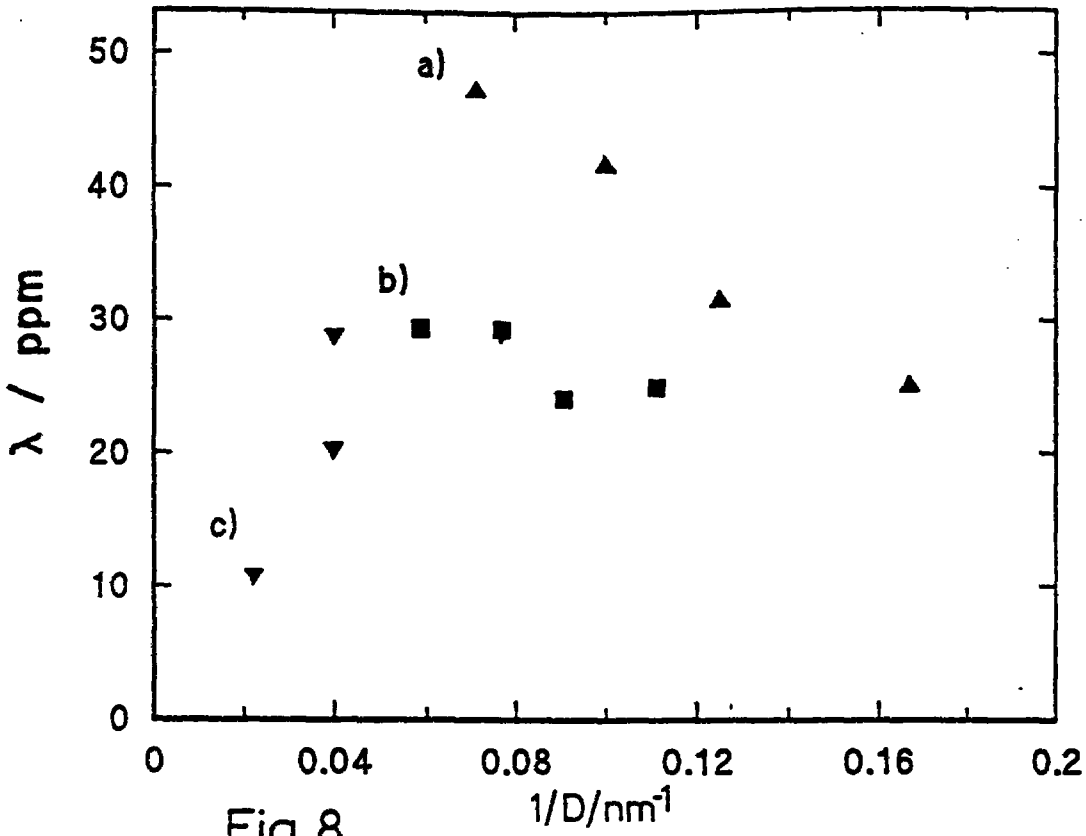


Fig.8

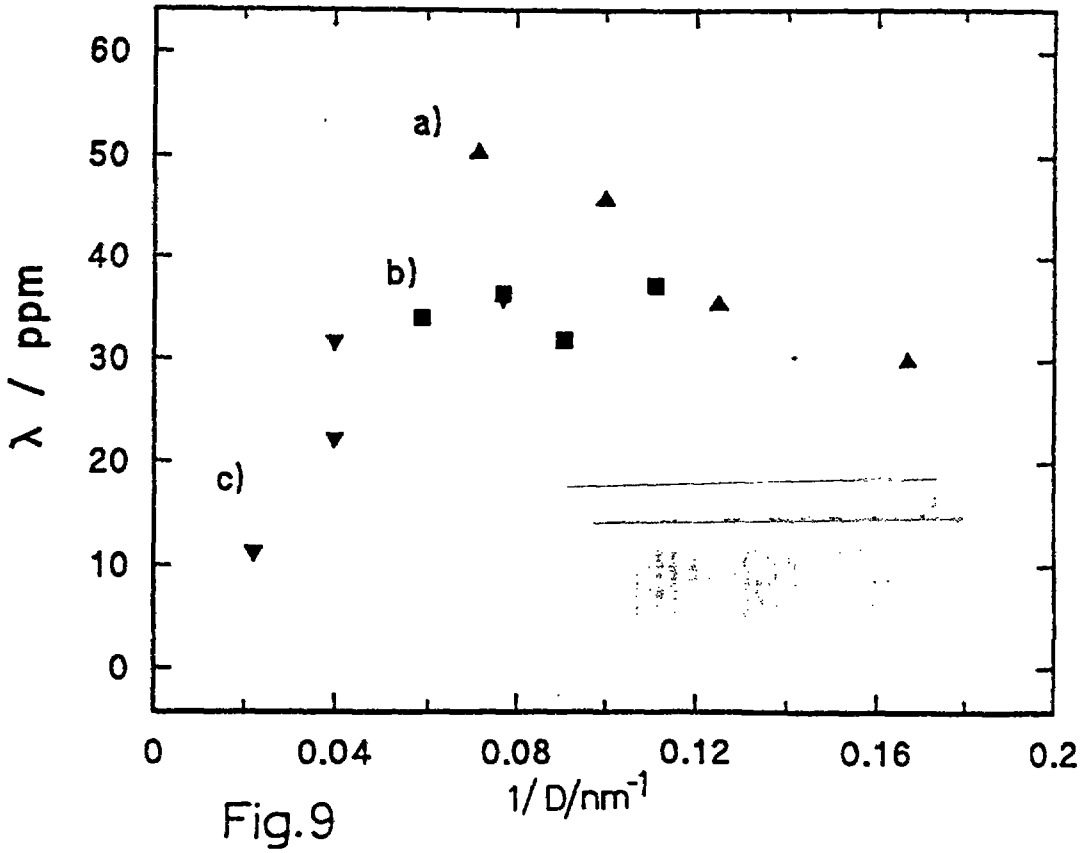


Fig.9

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	t(Fe)	t(Co)	t(Ag)		T_a	t_a
	(nm)	(nm)	(nm)			
S1	n		n	n = 1,2,3,4,5		
S2	n	2n	5	n = 1,2,5,10	290°C	30mins
S3	n	2n	5	n = 1,1.5,2,3	300°C	10-50mins
S4	n	2n	2	n = 1,1.5,2,3	300°C	10-50mins

Fig. 10

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MAGNETOSTRICTIVE MATERIAL

This invention relates to the production of materials with useful magnetostrictive properties.

Magnetostriction is the property which relates magnetic characteristics of a body of material to a change of the shape of the material. The property is seen in the change in size of bodies of certain materials when the magnetic environment changes or the change in magnetic characteristic when a force is applied to such a body to change its shape. Magnetostriction is a dimensionless quantity represented by the magnetostriction constant λ_s, relating magnetisation and shape change and in the SI system of units useful values are some tens or hundreds of parts per million, particularly for use in sensors and transducers. For such uses a high magnetomechanical coupling factor is desirable. Also "soft" magnetic properties are generally preferred.

While thin film amorphous alloys and magnetic multilayers individually provide some of the required properties there is still a strong need for a significant improvement in the properties available and for materials which exhibit a useful combination of such properties.

It is an object of the invention to provide materials with improved properties and methods of production of such materials.

In the publication "Magnetostriction of Multilayer Co/Ag Films" by H. Szymczak *et al.*, published at the International Magnetics Conference, April 17-20, 1990, Digest page HC-09, a study is made of cobalt and silver multilayers to sense the presence of any interdiffusion layers at the interfaces, which may cause changes in elastic and magnetoelastic properties of the multilayers, and the magnetostriction constant of the multilayers was measured against inverse layer thickness, but no evidence was presented or discussed to suggest a magnetostriction constant greater than that of naturally-occurring cobalt.

In the publication "Magnetostriction and In-Situ Measurement of Stress in Co/Pd Compositionally Modulated Multilayer Films During Fabrication", by H. Awano *et al.*, published at the International Magnetics Conference, April 17-20, Digest page HC-08, the anisotropy energy of the inverse magnetostriction effect in multilayer films is

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considered, and the stress at the interface of the films is measured, but it is stated that for a qualitative discussion of magnetostriction effect at the interface, further research is required.

According to the invention as claimed there is provided a magnetic material comprising at least two layers having different atomic structures, being at least one layer of a magnetically soft substance and at least one other layer of a second substance, the other layer having an atomic structure which cooperates with the atomic structure of the magnetically soft layer so as to alter the spacing of the atoms in at least the part of said magnetically soft layer adjacent the other layer whereby a magnetostrictive property of the material is increased, characterised in that the magnetically soft substance is immiscible with the other layer.

Optionally the alteration of the spacing is an increase in the spacing from the usual atomic spacing.

Usually, one layer of the material comprises a magnetic material and the other layer comprises either a magnetic or a non-magnetic material. Optionally the first and second layers comprise magnetic and non-magnetic forms of the same element. At least one of the layers may be an alloy.

The magnetically soft substance may be ferrous, such as Fe or an alloy between Fe and Co. The other layer may be of Ag. There may be a plurality of bilayers each comprising a layer of the first substance and a layer of the second substance, and optionally an insulator such as silicon oxide between bilayers.

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The magnetically soft substance may be chosen from the group of ferromagnetic elements of at least iron, nickel, cobalt, gadolinium and terbium.

The non-magnetic substance may be chosen from aluminium, magnesium, gold and silver and silicon oxide.

Preferably there are many first and second layers in the material forming a multilayer material. Generally there will be alternating said first and second layers. One first layer and the overlaid second layer may be identified as a bilayer. A first and second layer may have a third layer associated therewith as a trilayer and may further have a fourth layer associated therewith as a quadlayer. In general the material form can be expressed as of M multilayers each having N component layers. Typically N will be between two and five while M will have a value of a few tens, typically 20 to 50.

There may be a substrate on which the multilayer material is built up. The material may remain on the substrate for use or be released therefrom, for example by chemical means. The substrate may be glass or mica or KAPTON (RTM) or other suitable material. The magnetic material may be prepared in the place of eventual use, for example by deposition of successive component layers on a machine part or like object. There may be more than 30 bilayers.

Each layer of a bilayer may be at least a few nanometres thick, that is a few tens of atoms thick, and may be up to ten or more nanometres thick. It is believed that a well-defined boundary is useful where the second layer is overlaid on the first. This will produce a stress-free strain in the overlaid layer.

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One form of the material described above may prepared by a method including overlaying the second substance (component) on the first, providing a well-defined boundary where the second component is contiguous with the first, and causing or producing a stress-free strain in the overlaid component, at least adjacent the first component.

Another form of the material described above may prepared by a method including overlaying the second substance (component) on the first, permitting or causing the second component structure to be influenced by the first to produce said cooperation.

According to a particular aspect of the invention as claimed there is provided a magnetic material of a first component layer and a second component layer overlaid on the first, there being a lattice structure in at least one of said layers and in which magnetic material at least one lattice structure is modified where said first and second component layers are contiguous whereby the magnetostrictive property of the material is enhanced.

~~There may be an interface region where the layers are contiguous. The modification may be in the interface region. Where the interface region is in an elemental layer the lattice structure in at least said region may be one other than the usual one for the layer element.~~

According to the invention as claimed there is provided a method of producing a multilayer magnetic material including depositing a first component, causing or permitting a second component to be progressively overlaid on the first, controlling the depositing of the first component and the overlaying of the second component to produce respective different structures and causing or permitting the structures to cooperate to assist the magnetostrictive behaviour of the material.

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The method may include providing a first component layer of a first lattice structure, causing or permitting a second component layer of a second lattice structure to be progessively overlaid on the first, controlling the overlaying of the second component so that at least one component layer in a region contiguous to the interface of the layers has a lattice structure modified by the overlaying action.

The method may include overlaying by sputtering. The method may include overlaying substantially atom-by-atom. The method may include sputtering from a target which may be of one element or of two or more elements to enable the deposit of an alloy.

The method may include causing or permitting the respective different structures to be distinct and form an interface which provides a pseudo free surface.

A multilayer material as described above has an M/H loop characteristic which is altered by distorting the material, for example by bending.

According to a particular aspect of the invention as claimed there is provided a multiple multilayer magnetic material as described above in which the overlaid second layer is covered by an insulating layer, to enhance the frequency response.

According to the invention as claimed there is provided a magnetic material arrangement including a magnetic material as described above and means to cooperate with the material to convey to an external connection a response of the material to an external action.

The response may be a change of permeability or dimension and the external action may be the stressing of the material. The action may be flexure of a support for the material or an ambient pressure change, or an external magnetic field.

The arrangement may form part of an accelerometer, a torque sensor or like device.

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Embodiments of the invention will now be described with reference to the following examples and drawings, in which:

Figure 1 shows in schematic form a representation of a lattice structure of a multilayer material according to the invention,

Figure 2 shows in schematic form a representation of another lattice structure of a multilayer material according to the invention,

Figure 3 shows M/H characteristics of a material according to the invention,

Figures 4 and 5 show in schematic form a portion of a multilayer material, after deposition and after subsequent annealing respectively,

Figures 6 to 9 show plots of characteristics of materials embodying the invention,

Figure 6 shows magnetostriction constant of Fe/Ag multilayers (S1) versus 1/(modulation period),

Figure 7 shows magnetostriction constant of Fe-Co/Ag multilayers versus 1/(modulation period) calculated using E_f from both the volume average and the vibrating reed correction from equation 5, (squares and circles respectively). Also shown are the effective magnetostriction constants of the alloy layers alone, using the volume average E_f method (triangles),

Figure 8 shows magnetostriction constant of Fe-Co/Ag multilayers versus 1/(modulation period),

a) t_{Ag} = 2 nm (S4),

b) t_{Ag} = 5 nm (S3),

c) t_{Ag} = 5 nm, t_{anneal} = 30 minutes (S2),

Figure 9 shows magnetostriction constant of Fe-Co layers within Fe-Co/Ag multilayers versus 1/(modulation period), (a), (b) and (c) as in Figure 8, and

Figure 10 is a table of materials exemplifying the invention.

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Various proposals for multilayer materials having magnetic properties have been made, for example de Wit, Witmer and Dirne (Advanced Materials, Vol. 3 (1991) No. 7/8 pp 356 to 360) and Zeper, van Kesteren and Carcia (Advanced Materials, Vol. 3 (1991) No. 7/8 pp 397-399). In the first of these (de Wit, etc) a material with enhanced saturation magnetisation and relative permeability but minimal magnetostriction, specifically for a video recorder read/write head, is described. To achieve this a very small grain size is sought for the magnetic material layer (iron, grain size below 10 nanometres) and to produce such a grain size the iron layers are around 10 nanometres thick. The layers are separated by thinner layers of another ferromagnetic material, specifically an iron/chromium/boron layer. This layer needs to be at least two nanometres thick to prevent the grains in one iron layer linking with those in another iron layer by columnar growth and specifically epitaxial growth is not desired. In the second (Zeper et al) a magneto optical recording material is described, for enhanced recording density, which has adequate Kerr rotation and low enough Curie temperature while having a preferred magnetisation direction perpendicular to the material layer. This preferred direction only occurs with cobalt/platinum or cobalt/palladium multilayers when the cobalt layers are less than some 0.8 to 1.2 nanometres. The thickness of the non-magnetic but magnetically polarisable platinum or palladium layer is set by the required balance of Kerr effect and Curie temperature and for platinum is about 0.9 nanometres with a 0.4 nanometre cobalt layer. The cobalt layer is about two atom layers thick so that the required magnetic anisotropy is not reduced by "bulk" atoms between the surface layers. In Zuberek, Szymczak, Krishnan and Tessier (Journal de Physique, Vol. 12, No. 9, Colloque C8, December 1988, pp 1761 to 1762) it is suggested that a "bilayer" of evaporated component materials depends for

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effectiveness on the thinness of a nickel layer. In Dirne, Tolboom, de Wit and Witmer (J. Magn. Magn. Mat., No. 83, (1990) pp 399 to 400) the possibility of interface mixing in Fe/Co multilayers is discussed and seen as a disadvantage. Techniques to avoid this disadvantage are suggested.

Multilayers of the present invention as claimed do not follow the techniques of the above proposals. The present invention as claimed provides in one aspect that a layer of a first component overlaid on another layer, of a second component, is caused in a region contiguous with the other layer to adopt a structure other than that of the lattice which it would normally adopt. In this way, which can be described as lattice mismatch, a stress-free strain can be produced in at least the contiguous region of the overlaid layer. A result of this stress-free strain is an enhanced magnetostriction constant, λ_s.

Typically such layers of a first material overlaid on the second would be arranged as several bilayers on a substrate. Each layer of a bilayer would be some five to fifteen nanometres thick and deposited by sputtering so that atoms being deposited arrive at a rate slow enough to be able to be influenced in deposit position by the existing lattice of the second component layer or that of the first-deposited overlaid first component atoms. Care is needed to avoid building stress into the layers by the thermal effects of sputtering. Some ten to fifty bilayers can be deposited. The overlaid component applied adjacent to the second component lattice is conveniently caused to adopt a lattice spacing appropriate to that of the second component. This will cause a stress-free strain in at least the contiguous region of the overlaid component. The stress-free strain will be best exhibited if there is a sharp boundary between the layers.

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If required a pre-stress can be built in to the eventual material by, for example, keeping the substrate hot during deposition of components so that the substrate contracts on cooling. Also a magnetic bias can be built in by including a layer of permanently magnetisable component for example as or immediately above the substrate. A buffer substrate can also be used but care must be taken that the buffer transmits any strain that is applied for action by or on the magnetostrictive material. An inert or insulating layer may be applied to protect the material. Films may be grown under the influence of an imposed magnetic field to enhance the desired effect.

Figure 1 represents in a schematic manner the lattice at and near the surface of the second substance layer 12 and the lattice in the contiguous region of the overlaid substance layer 10. In the Figure overlaid substance atoms are indicated by the smaller, crossed, circles and the second layer substance atoms by the more widely spaced larger, open, circles. The lattice at 10A, furthest from the second substance layer 12, has the normal atomic spacing for the overlaid substance, while at levels 10B, 10C and 10D successively nearer the layer 12, the lattice of the overlaid substance is progressively more expanded.

To investigate the magnetostrictive properties further examples have been prepared. Typically these are a number of alternate layers of magnesium and iron, each a few nanometres thick and having a few tens of bilayers. The layers are not necessarily of the same thickness. The layers are deposited by sputtering from alternately exposed sources using conventional techniques. The preparation is arranged to ensure that bulk effects are not significant. The effect of stress on the film on the shape of the magnetisation/field (M/H) loop was also examined. A small amount of distortion, by bending the specimens, changed the loop shape by a detectable amount, increasing the value of M at lower values of H compared with the loop for the unstressed specimens.

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Other structures may be used to achieve the invention as claimed. Certain combinations of overlaid substance layers will produce a degree of alloying and this in turn can produce "dislocations" in an atomic level structure which would otherwise be adopted. Provided one substance layer is magnetic the other can be an insulator as the merging of the layers where they are contiguous can alter the spacing of the magnetic substance layer lattice and thus usefully alter the magnetostrictive property. Figure 2 shows in sketch form this action where, in a lattice 16, atoms of a magnetic substance, identified at m, are displaced at an interface 18 by the presence of atoms of another substance, identified at i, which may be non-magnetic even an insulator, in a lattice 14.

The present invention as claimed provides in another aspect an interface effect which can be a relatively sharp lattice mismatch as the overlaid components cannot easily mix.

An insulating layer, for example silicon oxide, can be introduced to produce an improvement in performance, especially an improved high frequency performance. The insulating layer thickness will be dependent on the frequency at which the device is to operate but will also be dependent on the effect of the relative thickness of the insulating layer and the bilayer on the propagation velocities in the insulating and magnetic components. From these considerations it is possible to derive suitable thickness relationships for a particular application of the multilayer material. In general the insulating layer thickness will be several times that of the individual magnetic component layer. In this way magnetostrictive materials with several tri- or quad- layers can be built up, of the general form of L multilayers each having N component layers. At least one of the N component layers will be a magnetic, generally a ferromagnetic, substance such as iron, nickel, cobalt, gadolinium or terbium while another will be a non-magnetic substance and may be a metal or an insulator, examples including aluminium, magnesium gold and silver and silicon oxide. If N is more than two one of the non-magnetic layers can be a metal and another an insulator. A further magnetic component layer may be included.

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A magneto-electric transducer can be formed by placing structures as exemplified above onto an electrostrictive structure such as PVDF, the dimensional changes in the magnetic structures imposing a strain on the electrostrictive structure, producing an electrical signal related to the action on the magnetostrictive structure.

Further examples of the invention will now be described.

Multilayers are grown using an rf glow discharge sputtering system in a sputter-up configuration with three 15 cm water-cooled magnetron target electrodes on a carousel, and a water-cooled substrate platen electrode which allows the substrate to be pre-sputtered. The substrate platen also has a heater capable of temperatures up to 600°C. The target-substrate distance was set at 60 mm, with an argon plasma pressure of two-thirds of a Pascal (5 mtorr). The sputtering equipment is a NORDIKO machine. The deposition rates were calibrated using a "Talystep" stylus based film thickness measurement instrument. Structural characterisation was also done using X-ray diffraction.

The substrates were glass and Kapton (Registered Trade Mark) (polyimide). The substrate for SAMR measurements (see below) was 25 mm thick Kapton, which was thin enough and had a low enough Young's modulus to avoid overly constraining the films while still being able to support them.

Fe/Ag multilayers were grown with 50 repeats (i.e. 50 bilayers).

Fe-Co/Ag multilayers (20-26, Figure 4) of 25 repeats (25 bilayers) were grown. The layers of Fe-Co alloy in the Fe-Co/Ag multilayers were produced by growing sandwiches 20-22-24 of Fe/Co/Fe between the silver layers 28 and 26. The multilayer was subsequently annealed to allow interdiffusion of the Fe and Co to form a layer 23 (Figure 5) of alloy of thickness t(Co)+2t(Fe) (the Ag is immiscible with both). The heat treatment was done in a non-inductively wound tube furnace, with the sample laid flat between two glass slides in a vacuum provided by a rotary pump and nitrogen trap to eliminate oxidation. The anneal time and temperature were 30 minutes and 290°C respectively.

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A series (S1) of five Fe/Ag multilayers was grown where t(Fe)=t(Ag)=1,2,3,4,5 nm.

A series (S2) of Fe/Co/Fe/Ag (i.e. Fe-Co/Ag) multilayers was grown where the thickness of the Co layer was twice that of each of the Fe layers so that the total thickness was the same for both, resulting in a post-anneal alloy of a nominal composition of $Fe_{50}Co_{50}$. The thickness of this layer was varied from 4 to 40 nm and t(Ag) was set at 5 nm. These were subsequently annealed in vacuum for 30 minutes at 290°C. Two other Fe/Co/Fe/Ag (i.e. Fe-Co/Ag) series were grown with silver layer thicknesses of 2 (S4) and 5 (S3) nm. The anneal times were longer for the films with thicker Fe/Co layers because of the greater degree of interdiffusion required. The anneal times (at 300°C) were 10, 20, 30 and 50 minutes for alloy layer thicknesses 4, 6, 8, 12 nm respectively.

As the layer thicknesses are decreased the volume fraction of the interface regions is increased. This increases the proportion of the multilayer material which is formed by atoms at or near the surface of a layer. The local moment is increased in the vicinity of a free surface so the local moment effect is enhanced as the interface volume fraction is increased. The texture of a layer is also significant and its

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retention can be enhanced by depositing the alloy directly and avoiding or reducing the annealing step. This will also encourage growth of the alloy in the [110] direction normal to the layer.

The coercive fields of Fe-Co/Ag multilayers are between 1 and 2 kAm^{-1}, reducing with layer thickness. Generally, the annealing rendered the multilayers magnetically softer. Also by annealing the multilayers were stress-relieved and the iron and cobalt layers were caused to mix more thoroughly. The interfaces with silver were probably sharpened, since silver is immiscible with iron and with cobalt. For the thinnest iron and cobalt layers (D=9nm, i.e. t_{Co}=2nm) the region between silver layers would have been almost completely alloyed as-deposited [Dirne and Denissen, 1989]. Annealing this multilayer increased (approximately doubling) its coercive field in distinction to the other multilayers, and it became impossible to measure the magnetostriction. This is considered below.

The length of an anneal required to achieve complete alloying at a given temperature will depend on the thickness of the component layers. Thus it is possible that the anneals did not completely alloy the layers, especially in the thicker multilayers, and that they were longer than required for the thinner multilayers. The optimum heat treatment may not have been used.

Magnetostriction measurements were made using the Small Angle Magnetisation Rotation method proposed by Norita et al. [1980] and widely used on amorphous ribbons.

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The basic principle of the method is as follows: the sample to be measured is saturated in the longitudinal direction (z) by a large dc field. A smaller ac field of frequency ω acts in the transverse direction (y). This results in a small degree of rotation of the macroscopic moment direction about the longitudinal direction. If a stress, σ, is also applied to the sample along this direction, the angle of moment rotation is given by

$$\Theta = \frac{\mu_o M_s H_y}{H_z + H_d + H_\sigma} \qquad (1)$$

where H_d is the demagnetising field due to the sample shape, and H_σ is the stress-induced anisotropy field. This is given by

$$H_\sigma = \frac{3\lambda_s \sigma}{\mu_o M_s} \qquad (2)$$

The sample is mounted within a search coil whose axis is in the longitudinal direction. Thus a rotation of the magnetisation about this axis results in an induced ac voltage across the search coil with frequency 2ω, $V_{2\omega}$.

A change in the stress on a magnetostrictive material effects a change in the stress induced anisotropy field. In the case of a positively magnetostrictive material, a tensile stress tends to make the stress axis magnetically easier, so aligning the moments towards this axis. This would cause the angle of moment rotation to decrease, so decreasing the search coil output voltage.

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This change in output voltage can be compensated by adjustment of the dc field, H_z. The change in H_z is then equal to the change in H_σ. Thus, the magnetostriction constant, λ_s, is generally determined by plotting the change in H_z for a series of applied stresses, so that (where moment rotation is small)

$$\lambda_s = \frac{\mu_o M_s}{3} \frac{dH_z}{d\sigma_a} \Big| V_{2\omega}, H_y \quad . \tag{3}$$

The multilayers are too thin to be self supporting and need to be measured while still on their substrates. Although these can have elastic moduli far lower than the multilayers, they are much thicker and so accommodate a significant proportion of the stress, so it is necessary to correct for their presence.

This correction can be made by assuming that the multilayer is strongly adhered to the substrate so that they strain by the same amount and that the Young's modulus, E, of the whole is equal to the volume average of the component layers and the substrate. This assumes that the reed as a whole (i.e. substrate + multilayer) is effectively homogeneous. In this case the magnetostriction is given by

$$\lambda_s = \frac{\mu_o M_s}{3} \left[\frac{E_{av} A_{f+s}}{E_f} \right] \frac{dH_z}{d\sigma_a} \Big| V_{2\omega}, H_y \quad . \tag{4}$$

The same method of correction was also used by Wenda et al. [1987] on measurements of FeB and FeSiB single films sputtered onto polyester substrates.

Measurements of multilayer samples were taken on two slightly different SAMR apparatus arrangements. The dc field was applied using a solenoid, while the ac field was applied with a pair of Helmholtz coils. The sample was placed within a search coil (4000 turns) coaxially with the solenoid, in the centre of the Helmholtz coils. In the second configuration, a second, "bucking", coil was also present and was backed off

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against the search coil to cancel out any induced voltages common to both, i.e. due to sources other than the sample magnetisation.

The sample was clamped at both ends. The clamp at one end was fixed while that at the opposite end was connected to a cable which was loaded with known weights. The dc field was determined from the product of the current and the number of turns per unit length in the solenoid.

The values of B_s were obtained from literature, namely 2.0T for iron and 2.3T for $Fe_{50}Co_{50}$ alloy. The value of B_s for the whole multilayer was simply B_s for the relevant magnetic layer multiplied by its volume fraction.

The Young's modulus of the multilayers was measured using the vibrating reed method, which is fully described in Berry and Pritchet [1975]. It is based on the measurement of the resonant frequencies of various modes of cantilever vibration, f_n, of reeds clamped at one end.

Reeds were cut from the multilayer using a sharp blade and had free lengths and widths of approximately 13-20 mm and 1 mm respectively.

The value of E for the multilayer can be obtained from the value for the reed from the expression (from Berry and Pritchet):

$$\frac{f^2_{reed}}{f^2_{substrate}} = 1 + \frac{t}{d}\left[\frac{3E_f}{E_s} - \frac{\rho_f}{\rho_s}\right], \qquad (5)$$

where the subscripts f and s refer to the multilayer and substrate respectively, and t and d are the multilayer and reed thicknesses (t«d).

The value of λ_s for the multilayer was calculated from equation 4. In the case of the Fe-Co/Ag multilayers (t(Co)=2t(Fe)), two data sets are presented, corresponding to the two methods of calculating E_f. For the Fe/Ag data, only the values of λ_s derived from the assumption of a volume average E_f are shown.

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The data for λ_s is shown versus the reciprocal of the modulation period, D, in Figure 6. At 1/D=0 the value of λ_s should be that for bulk iron. The bulk value of λ_s of polycrystalline iron is -7 ppm, although this becomes more positive as it becomes more textured, so that perfectly textured polycrystalline iron with the [110] direction normal to the film surface has a value of λ_s of approximately 14 ppm. In practice, the measured value of λ_s for a single iron film will generally lie between these two values. As a check a thick (0.8 μm) iron film was deposited onto each side of a Kapton susbtrate (1.6 μm total). λ_s was measured to be -3.0 ppm.

As D decreases so that the volume fraction of the Fe/Ag interface layers increases, it is found that λ_s becomes more positive. Hence the presence of silver is seen to enhance the value of λ_s in iron, as expected.

A series of multilayers was grown with layers of Fe and Co of equal thickness (5 nm) with silver layers of varying thickness. Because there was approximately twice the amount of iron as cobalt in the alloy layer after annealing, it is probable that there was a composition gradient across the layer, with a corresponding variation in λ_s. The λ_s data from this series of multilayers did not show any systematic trend and there was a wide scatter in the data and is therefore not shown here.

The λ_s data for the second series of multilayers is shown for these films versus the reciprocal of the modulation period in Figure 7. Also shown is the effective λ_s of the magnetostrictive material alone, derived via the data assuming a volume average Young's modulus. There are pairs of data points obtained via the Berry and Pritchet E_f correction method for most samples, corresponding to two values of E_f obtained from the third and fourth modes of reed vibration. There are two samples measured with t(alloy)=30 nm since two sections

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were taken from the same substrate and were annealed independently. However, E_{reed} for this sample was only measured using a reed from one of the anneals, so the pair of points from this sample with the higher λ_s values may be subject to large error. This figure illustrates the difference in the data resulting from the two methods of correction of the Young's modulus.

Before anneal, most samples were found to have small negative magnetostriction (generally smaller than -5 ppm), and large positive λ_s after anneal confirming that alloying of the component TM layers had alloyed. The sample with t(alloy)=4 nm gave a large positive λ_s before annealing, indicating that the iron and cobalt layers had intermixed to a high degree. It is suggested in Dirne and Denissen [1989] that Fe-Co alloys form at interfaces during deposition, with thicknesses of approximately 1 nm This means that approximately 50% of the Fe-Co in this sample was intermixed before anneal. Unfortunately, no SAMR response could be obtained from the sample after anneal. As stated earlier, this was accompanied by a remarkable increase in coercive field. It may be that since the layers were so thin, the anneal caused disruption of the coherency of the multilayer.

From the volume average data, it is seen that λ_s of the multilayer increases to approximately 20 ppm as the alloy thickness is reduced to 8 nm, equivalent to a value of λ_s of about 36 ppm for the alloy layers.

The magnetostriction of all three series (S2, S3, S4) of the Fe-Co/Ag multilayers were measured and are shown in Figure 8. These values were calculated using the volume average Young's moduli. The corresponding values of λ_s for the alloy alone are shown in Figure 9. A clear trend is seen for the series of multilayers (S4) with t_{Ag}=2 nm, where λ_s decreases with decreasing alloy layer thickness. The data for the series of multilayers with t_{Ag}=5 nm also show some

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reduction of λ_s with t_{Fe-Co}, but in Figure 8, the values of λ_s for the alloys are approximately constant (within error), indicating that the reduction in λ_s for the multilayer is due to loading by the silver layers. The reduction in λ_s for the t_{Ag} data set cannot be accounted for in this way.

The decrease of λ_s of the thicker multilayers from the series (S2) is thought to be due to the iron and cobalt layers not fully intermixing during the half-hour anneal. It should be noted that the values of λ_s for the two multilayers comprising layers of 2 nm Fe, 4 nm Co and 5 nm Ag, annealed at approximately 300°C for 30 minutes were measured to be the same within error. This adds confidence to the repeatability and accuracy of the data. If intermixing is poor composition gradients can arise.

The value of λ_s of the alloy layer was measured to be approximately 35±3 ppm. This compares with approximately 80 ppm for the bulk. A multilayer of Fe/Co was deposited without silver layers, and was annealed nominally to form a single layer (0.5 μm total thickness) of Fe-Co alloy, T_a=300°C, t_a=30 minutes. The value of λ_s of the film was measured to be 43 ppm. A further heat treatment of 30 minutes at 300°C was found to cause an increase in λ_s to approximately 65 ppm.

The magnetostriction constant obtained from SAMR values depends on the Young's modulus of the reed and also on that of the film only. Discrepancies have arisen between the values of E_{film} corrected from E_{reed} from two different methods of obtaining this correction. The method assuming a volume average of the moduli is thought to be the more reliable.

The values of λ_s of the Fe/Co/Fe/Ag multilayers were small and negative, as would be expected from an average of Fe and Co. After anneal, the λ_s values were large and positive, indicating that the Fe and Co had alloyed to a large extent. Annealing also had the effect of making the films softer, predominantly due to relief of internal stresses. Values of H_c of the post-anneal samples were typically of the order of 1kA/m.

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Figure 3 shows the effect of stress on the M/H loop for one of the multilayers from series S4. The specific one used is that where n = 3. The multilayer had 25 bilayers, each deposited as 3 nm Fe/6 nm Co/3 nm Fe/2 nm Ag, supported on a 25 micrometre Kapton layer. Annealing was 50 minutes at 300°C under vacuum. The dotted line is the M/H characteristic at stress $\sigma = 0$. When a stress of 110 MPa is applied the shape and size of the M/H loop changes very significantly, shown in the solid line. M is in arbitrary units.

In the case of the Fe/Ag multilayers, reduction in the thickness of the layers (i.e. an increase in the volume fraction of the interface regions) has the effect of making the magnetostriction more positive. The measured values of λ_s in this study increased from approximately -0.3 ppm for 5 nm Fe/5 nm Ag to approximately +3 ppm for 1 nm Fe/1 nm Ag. This is consistent with an increase in the local magnetic moment of iron at the Fe-Ag interfaces.

In the case of the Fe-Co multilayers, λ_s was seen to decrease with decreasing modulation period, where this was shown to be due to mechanical loading by the silver layers, where t_{Ag}=5 nm. When this correction was made, the value of λ_{Fe-Co} were found to be approximately 35 ppm, which is considerably lower than those of bulk Fe-Co alloys. Non-optimal heat treatments are thought to contribute to the reduction in the values, as indicated by the decrease in λ_s for the thicker multilayers annealed for 30 minutes. A definite decrease in λ_s with decreasing alloy layer thickness was seen for the multilayers where t_{Ag}=2 nm, which cannot be accounted for by the loading by the silver layers. The reason for this is not clear. It is possible that the silver layers are so thin that the alloy layers have percolated through, especially where these are appreciably thicker than the silver, so that the parameters of the multilayer approach those of a bulk material. It may be that inclusion of silver in the multilayer causes a reduction in λ_{Fe-Co}.

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It is seen from the above that thickness and crystallographic texturing of the alloy layer and silver layer loading all can affect the value of λ_s. Those skilled in the art will be able to proceed on the basis of the techniques set out above to produce multilayers with significant values of λ_s using appropriate component layers and dimensions.

Some of the above examples show that the technique of causing a magnetic component layer to adopt, in a surface region at least, a lattice structure other than the one it would normally adopt produces a magnetic material which exhibits a non-zero magnetostrictive coefficient, albeit at a coercivity more appropriate to a "hard" magnetic material than a "soft" one, but noting that this parameter was not controlled.

Others of the above examples show that the technique of causing a sharp lattice mismatch in overlaid component layers produces a magnetic material which exhibits a non-zero magnetostrictive coefficient. In particular a sharp lattice mismatch, such as occurs with immiscible component layers, can produce a pseudo free surface. The term pseudo free surface is used to describe the arrangement where the surface of a thin layer can emulate a free surface although another layer is contiguous.

The magnetic material can be used in sensing devices, such as pressure gauges, accelerometers and torque sensors, by attaching the material to a structure or by depositing the material in the place where it is to be used.

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CLAIMS

1. A magnetic material comprising at least two layers (10, 12) having different atomic structures, being at least one layer (10) of a magnetically soft substance and at least one other layer (12) of a second substance, the other layer (12) having an atomic structure which cooperates with the atomic structure of the magnetically soft layer (10) so as to alter the spacing of the atoms in at least the part (10D) of said magnetically soft layer (10) adjacent the other layer (12) whereby a magnetostrictive property of the material is increased, characterised in that the magnetically soft substance is immiscible with the other layer.

2. A material according to claim 1 characterised in that the alteration of the spacing is an increase (10D) in the spacing from the usual atomic spacing (10A, 10B).

3. A material according to claim 1 or 2 characterised in that at least one of the one and other layers (23, 26) comprises an alloy (23).

4. A material according to claim 3 characterised in that the one and the other layer comprise magnetic and non-magnetic forms of the same element.

5. A material according to any preceding claim, wherein the magnetically soft substance (10) is ferrous.

6. A material according to claim 5, wherein the ferrous substance is Fe or an alloy between Fe and Co.

7. A material according to any preceding claim, wherein the said other layer (12) is of Ag.

8. A material according to any preceding claim, characterised in that there is a plurality of bilayers (10, 12), each bilayer comprising a layer (10) of a magnetically soft substance and a layer (12) of the second substance.

9. A material according to claim 8, further comprising an insulator between bilayers.

10. A material according to claim 9, wherein the insulator is silicon oxide.

11. A method of preparing a magnetic material according to any preceding claim, characterised by comprising preparing a layer (12) of a first substance and overlayering a layer (10) of the second substance on the first layer whereby the atomic structure of the second layer is altered by the atomic structure of the first layer at least in the part of the second layer (10D) adjacent the first layer (12), and a magnetostrictive property of the

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material is increased.

12. A method according to claim 11 characterised in that at least the step of overlayering comprises a sputtering process.

13. A method according to claim 12 characterised in that at least the step of overlayering comprises an atom-by-atom deposition process.

14. A method according to any of claims 11 to 13 characterised by further comprising the steps of overlayering the layer of the second substance with further consecutive layers of the first and second substances, the steps being such that the atomic structure of at least each second layer is altered by the atomic structure of the previously-overlayered first layer.

15. A method according to any of claims 11 to 13 characterised by further comprising the steps of overlayering the second layer (22) with a further layer of the first substance (24), the first and second substances being capable of forming an alloy; overlayering the further layer of the first substance (24) with a layer of a third substance (26); and annealing the magnetic material so that the first and second layers form a layer of an alloy (23) on the layer of the third substance (26).

16. A method according to any of claims 11 to 13 characterised in that the layer of the first substance is prepared on a substrate.

17. A method according to claim 16 characterised in that the substrate is a machine part.

18. A magnetostrictive device comprising a magnetostrictive component and sensing means to sense a magnetostrictive change in said component in response to an external physical effect, characterised in that said component is a magnetic material which is according to any of claims 1 to 10.

19. A magnetostrictive device according to claim 18 in which the sensing means senses a change in magnetic permeability of the magnetostrictive component.

20. A magnetostrictive device according to claim 18 in which the sensing means senses a change in a dimension of the magnetostrictive component.

21. A magnetostrictive device according to claim 18 which is an accelerometer.

22. A magnetostrictive device according to claim 18 which is a torque sensor.

23. A magnetostrictive device according to claim 18 which is a pressure gauge.

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MAGNETOSTRICTIVE MATERIAL

ABSTRACT

A magnetic material of at least two component parts arranged to have respective structures which mutually are not homogenous, the structure of one part cooperating with the structure of the other to assist (fig. 3) the magnetostrictive behaviour of the material. The cooperation can be the structure of one part altering the atomic spacing of the other part (fig. 1, fig. 2). The alteration can be an increase from the usual atomic spacing. There can be contiguous component parts and the cooperation can be a pseudo free surface on at least one said part or a reduced restriction of the local moment otherwise achieved by the contiguous parts (fig. 5). A component can be at least partly an alloy can have ratio other than the usual one for said alloy. One of the components can be non-magnetic.

FIGURE 2.

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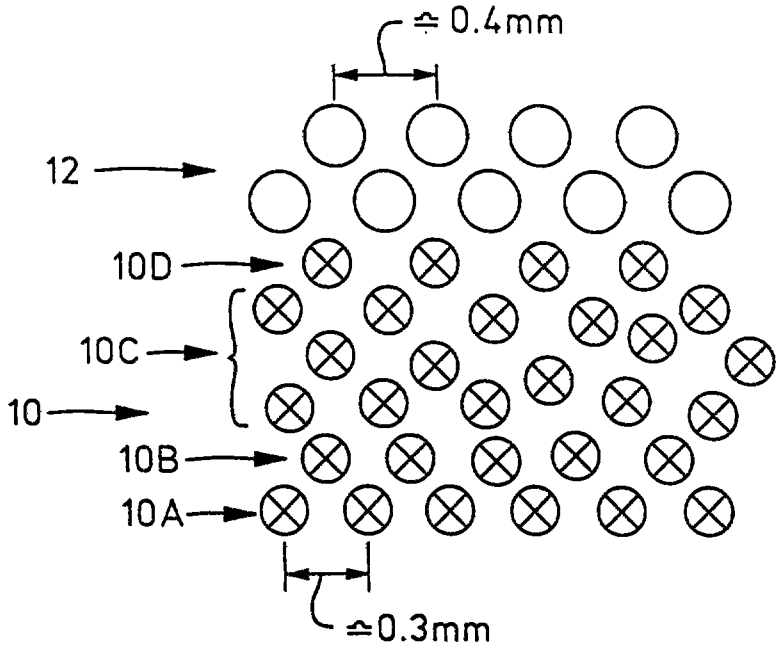


Fig.1

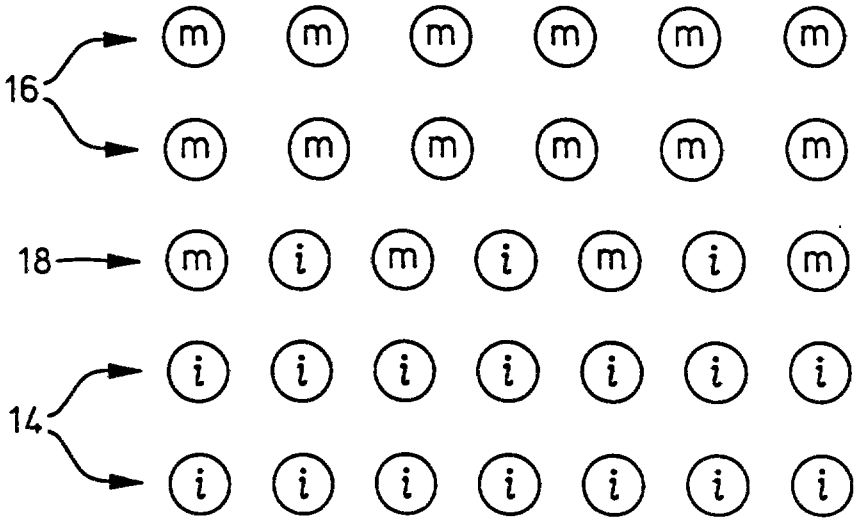


Fig.2

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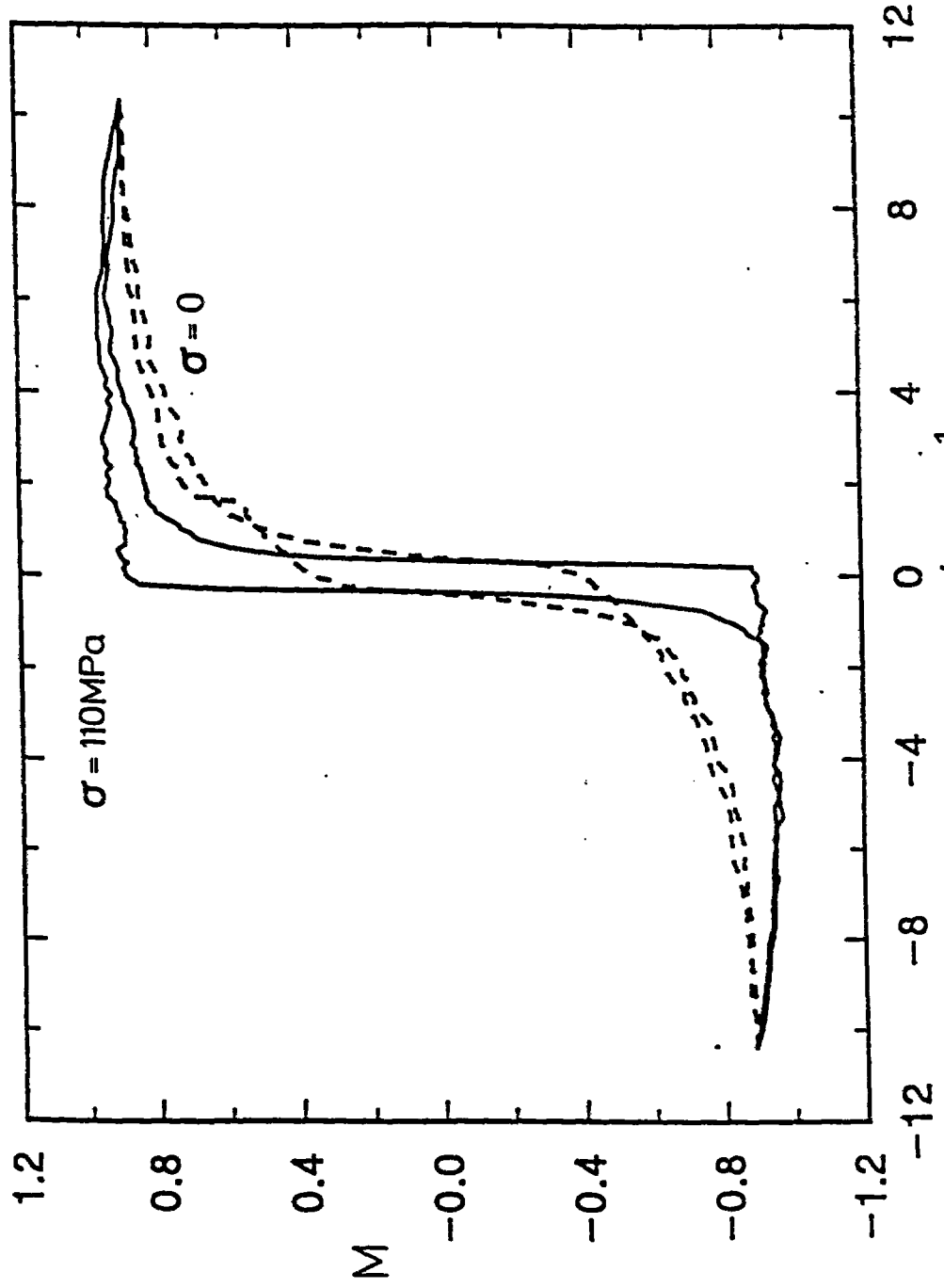


Fig. 3

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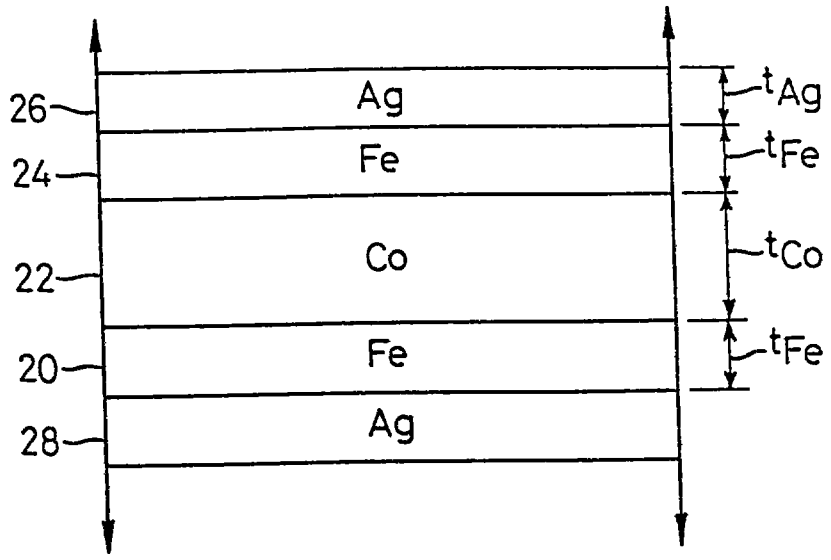


Fig. 4

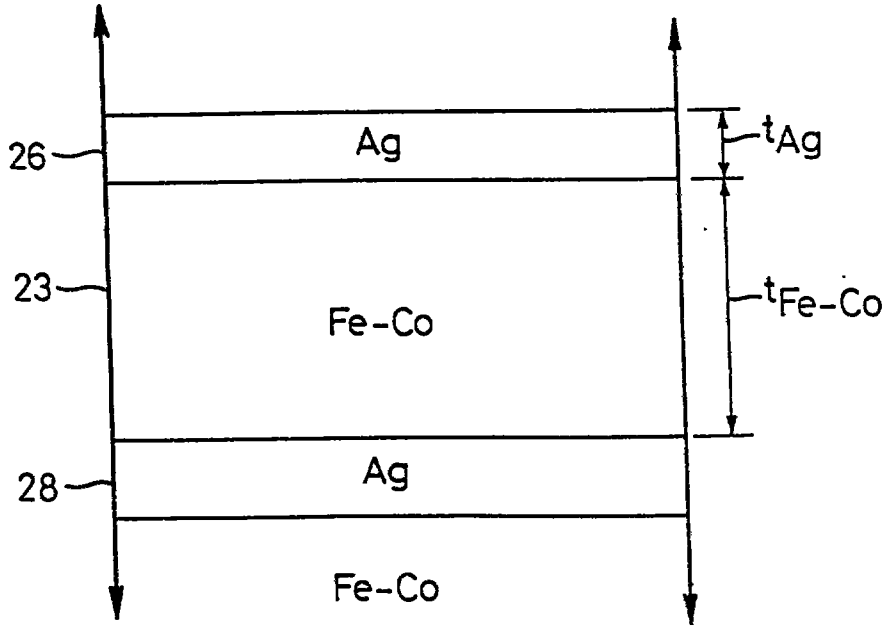


Fig. 5

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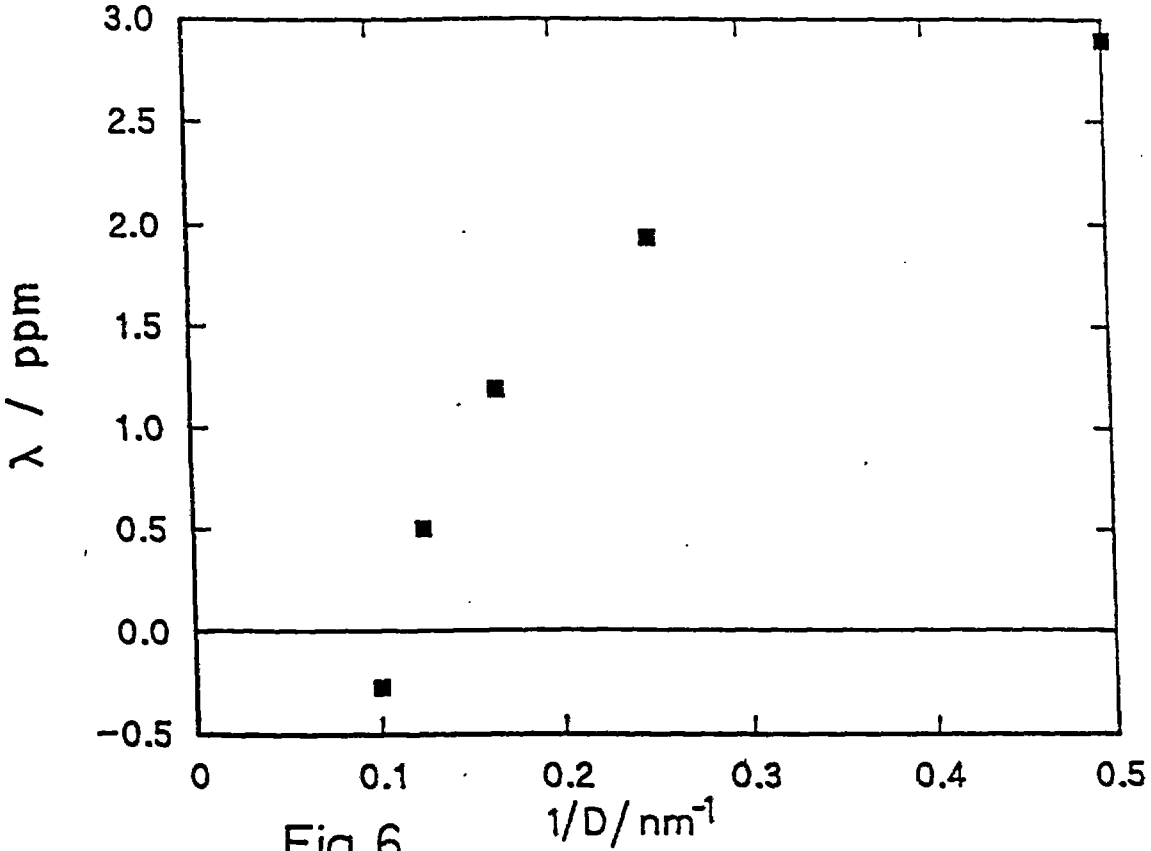


Fig.6

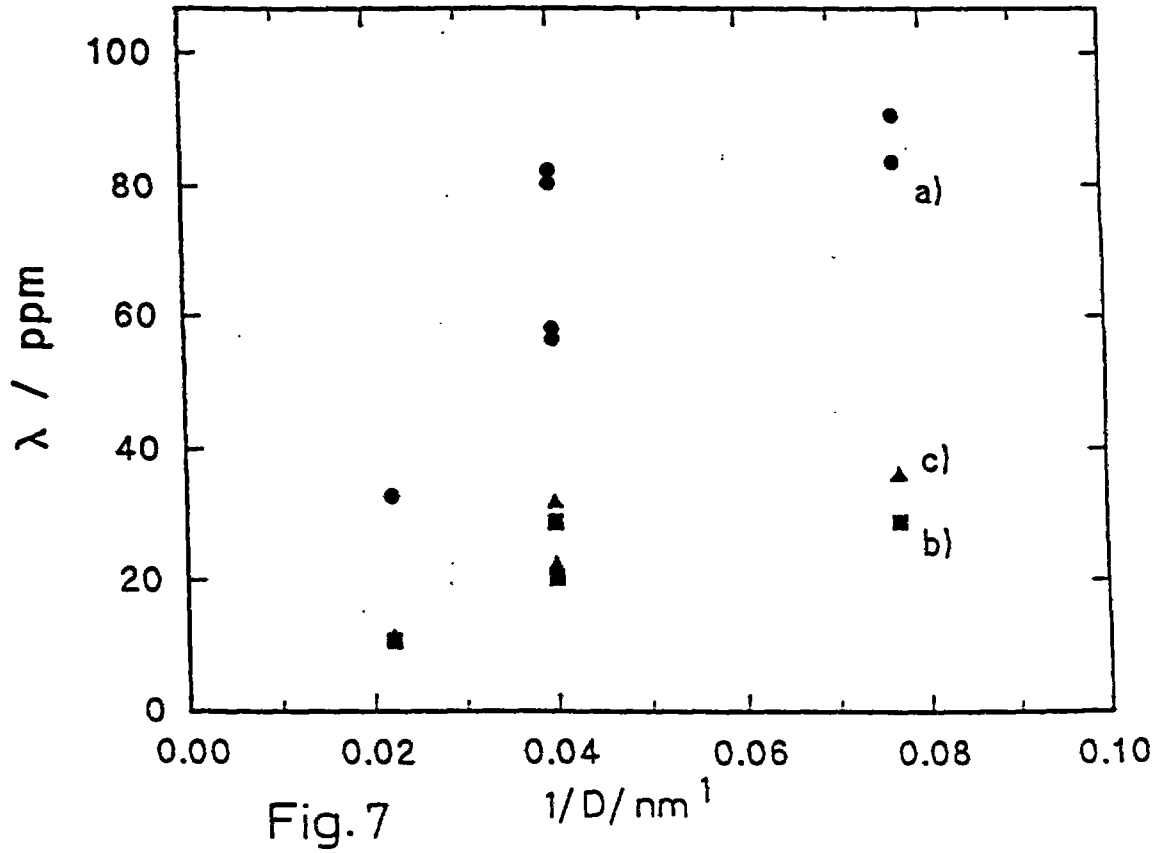


Fig. 7

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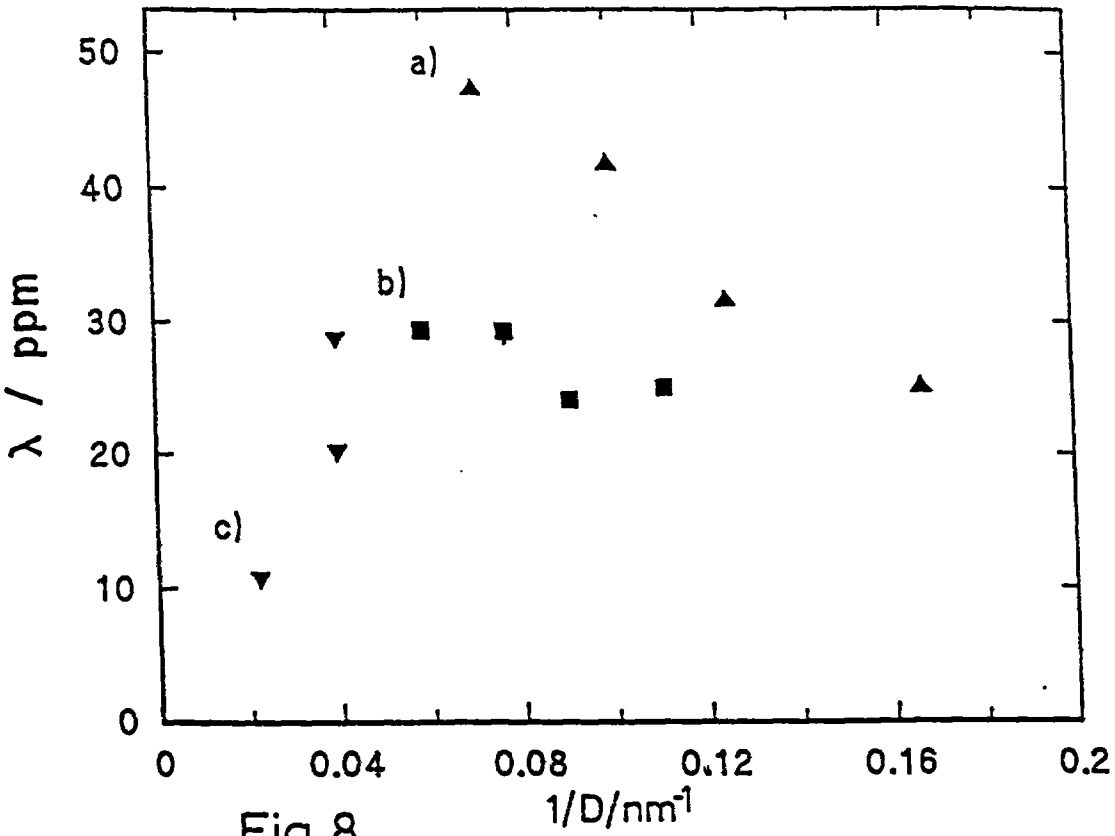


Fig.8

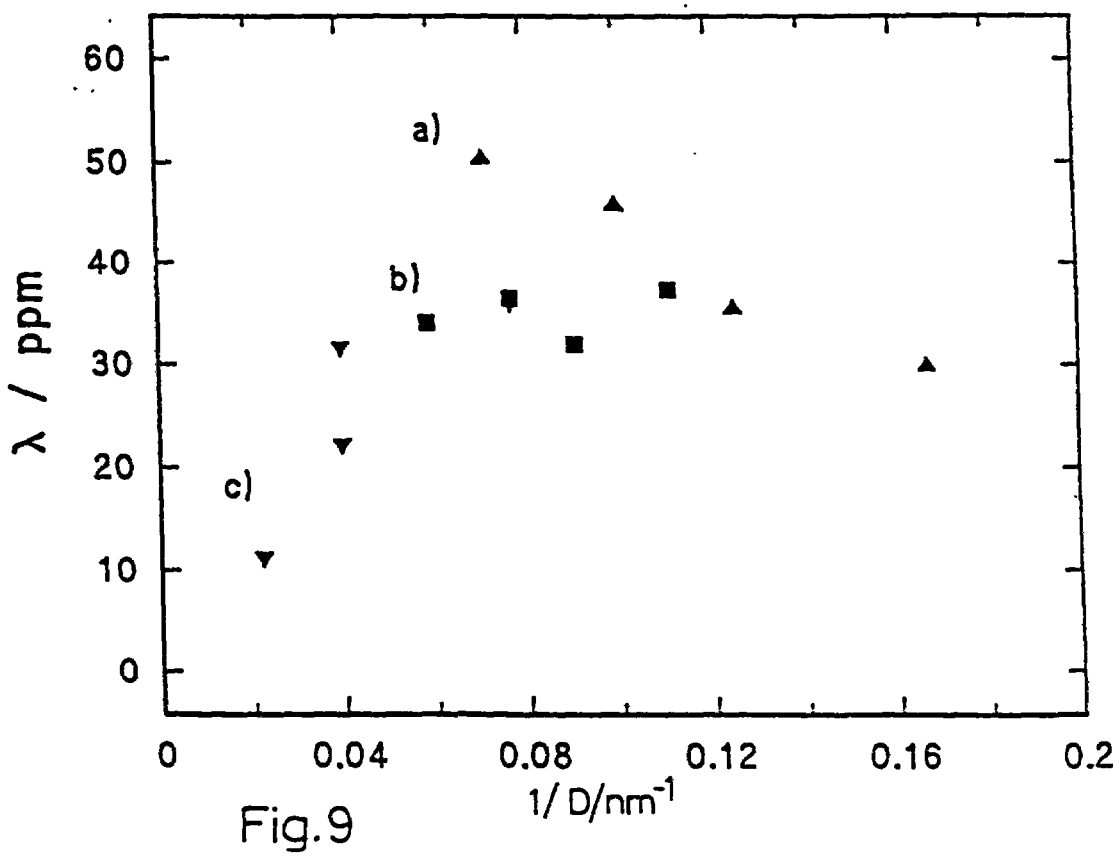


Fig.9

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	t(Fe)	t(Co)	t(Ag)		T_a	t_a
	(nm)	(nm)	(nm)			
S1	n		n	n = 1,2,3,4,5		
S2	n	2n	5	n = 1,2,5,10	290°C	30mins
S3	n	2n	5	n = 1,1.5,2,3	300°C	10-50mins
S4	n	2n	2	n = 1,1.5,2,3	300°C	10-50mins

Fig. 10