

Fe-Co ALLOY SPUTTERING TARGET MATERIAL AND METHOD FOR PRODUCING  
SAME, AND SOFT MAGNETIC THIN FILM LAYER AND PERPENDICULAR MAGNETIC  
RECORDING MEDIUM USING SAME

5

## TECHNICAL FIELD

[0001]

The present invention relates to Fe-Co alloy sputtering target materials, methods for producing such materials, thin soft magnetic layers, and perpendicular magnetic recording media including such layers.

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## BACKGROUND ART

[0002]

Recent advances in magnetic recording technology are remarkable, and efforts to provide drives with higher capacities have been directed to the development of high-density magnetic recording media. Accordingly, perpendicular magnetic recording has been put to practical use, which provides higher recording densities than conventional in-plane magnetic recording media. Perpendicular magnetic recording, which is suitable for high recording density, uses a perpendicular magnetic recording medium including a magnetic film having an easy magnetization axis oriented perpendicular to the surface of the medium. For perpendicular magnetic recording, double-layer recording media have been developed, each including a magnetic recording layer with enhanced recording sensitivity and a soft magnetic layer. The magnetic recording layer is typically composed of a CoCrPt-SiO<sub>2</sub> alloy.

[0003]

JP2006-294090A (PTL 1) discloses a Fe-Co alloy film serving as a soft magnetic layer. In PTL 1, Si, Ni, Ta, Nb, Zr, Ti, Cr, and/or Mo is added to Fe and Co in an amount of 20 at.% or more to form an amorphous or microcrystalline film structure.

[0004]

JP2010-18884A (PTL 2) discloses a Fe-Co alloy sputtering target material having a composition (Fe - 20 to 80 Co) - 4 to 25 Nb or Ta and having a eutectic structure composed of intermetallic compound phases with a size (largest inscribed circle) of 10  $\mu\text{m}$  or less.

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## CITATION LIST

### PATENT LITERATURE

[0005]

PTL 1: JP2006-294090A

10 PTL 2: JP2010-18884A

## SUMMARY OF INVENTION

[0006]

The formation of the above Fe-Co alloy film requires a corresponding Fe-Co sputtering target material. However, a sputtering target material designed to achieve the above film composition readily cracks during sputtering. In particular, the sputtering target material disclosed in PTL 2, which has the composition (Fe - 20 to 80 Co) - 4 to 25 Nb or Ta and has a eutectic structure, readily cracks during sputtering.

[0007]

20 The inventors, who have conducted intensive research to overcome the foregoing problem, have discovered that the cracking of a sputtering target material during sputtering can be prevented by controlling the microstructure of the sputtering target material, which has led to the present invention. Specifically, the inventors have discovered that a Fe-Co alloy sputtering target material that does not crack during sputtering can be provided by controlling the area ratio of the eutectic structure in the overall microstructure to 30% or less.

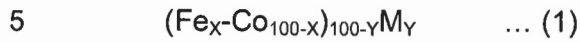
[0008]

Accordingly, it is an object of the present invention to provide a Fe-Co alloy sputtering

target material that does not crack during sputtering.

[0009]

According to an aspect of the present invention, there is provided a Fe-Co alloy sputtering target material composed of a Fe-Co-M alloy represented by formula (1):



where the atomic ratios are  $0 \leq X \leq 100$  and  $4 \leq Y \leq 28$ , and the element M includes at least one element selected from Nb, Ta, Mo, W, Cr, and V. The sputtering target material has a microstructure including a phase based on Fe and Co and an intermetallic compound phase containing the element M and at least one of Fe and Co. The area  
10 ratio of a eutectic structure in the overall microstructure is 30% or less.

[0010]

According to another aspect of the present invention, there is provided a method for manufacturing the Fe-Co-M alloy sputtering target material of the present invention. This method includes the step of consolidating an alloy powder prepared by gas atomization of  
15 a melt of the alloy by hot isostatic pressing (HIP).

[0011]

According to still another aspect of the present invention, there is provided a thin soft magnetic layer deposited from the Fe-Co-M alloy sputtering target material of the present invention.

20 [0012]

According to a further aspect of the present invention, there is provided a perpendicular magnetic recording medium including the thin soft magnetic layer of the present invention.

## BRIEF DESCRIPTION OF DRAWINGS

25 [0013]

[Fig. 1] Fig. 1 is a set of schematic views illustrating eutectic structures according to the present invention, where  $s$  is the lateral size of intermetallic compound phases, and  $d$  is the closest distance between the intermetallic compound phases.

[Fig. 2] Fig. 2 is an electron micrograph showing a reflected electron image of No. 1 in Table 1.

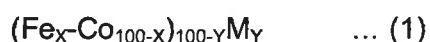
## DESCRIPTION OF EMBODIMENTS

5 [0014]

A Fe-Co alloy sputtering target material of the present invention will now be described in detail. The symbol "%" (percent)" indicating the content of each constituent refers to at.% (atomic percent) unless otherwise specified.

[0015]

10 The Fe-Co alloy sputtering target material of the present invention is composed of a Fe-Co-M alloy represented by formula (1):



where the atomic ratios are  $0 \leq X \leq 100$  and  $4 \leq Y \leq 28$ , and the element M includes at least one element selected from Nb, Ta, Mo, W, Cr, and V. The sputtering target material  
15 has a microstructure including a phase based on Fe and Co and an intermetallic compound phase containing the element M and at least one of Fe and Co. The area ratio of a eutectic structure in the overall microstructure is 30% or less.

[0016]

In formula (1), X is 0 to 100, preferably 20 to 80, even more preferably 25 to 75. That is,  
20 X includes 0, indicating that the sputtering target material contains at least one of Co and Fe.

[0017]

In formula (1), the element M includes at least one element selected from Nb, Ta, Mo, W, Cr, and V. These elements are metals that form a eutectic structure with Fe and/or Co;  
25 therefore, the sputtering target material contains at least one of these elements. In addition to Nb, Ta, Mo, W, Cr, and V, addition of less than 4% Al, Si, B, Ni, Mn, Cu, Ti, and/or Zr is allowable in the present invention, which does not interfere with the advantages of the present invention.

[0018]

In formula (1), Y is 4 to 28, preferably 10 to 25, even more preferably 15 to 23. The element M is a metal that forms a eutectic structure with Fe and/or Co; therefore, the sputtering target material contains at least one of these elements. If Y is less than 4,  
 5 however, the effect of addition is insufficient. If Y is more than 28, the sputter target readily cracks during sputtering.

[0019]

The area ratio of the eutectic structure in the overall microstructure is 30% or less, preferably 15% or less, more preferably 8% or less. The maximum area ratio of the  
 10 eutectic structure is limited to 30% because, if it exceeds 30%, the sputter target readily cracks during sputtering. To control the area ratio of the eutectic structure to 30% or less, consolidation may be performed at a consolidation temperature of 970°C or higher, preferably 1,000°C or higher, and a consolidation pressure of 130 MPa or higher.

[0020]

15 The area ratios of the above eutectic structure and the residual structure may be determined as follows. A specimen for scanning electron microscopy (SEM) is sampled from a scrap of the sputtering target material. A cross section of the specimen is polished, and ten reflected electron images are captured at a magnification of 2,000 times. The eutectic structure and the residual structure are extracted through image processing,  
 20 and the area (S1) of the eutectic structure, the area of the residual structure, and the area (S2) of the examination field of view are measured by image analysis. The area (S1) ratio of the eutectic structure in the area (S2) of the examination field of view is calculated from  $S1/S2 \times 100$  (%).

[0021]

25 Fig. 1 is a set of schematic views illustrating example eutectic structures according to the present invention. Fig. 1(a) is a top sectional view, Fig. 1(b) is a side view, and Figs. 1(c) and 1(d) illustrate eutectic structures having different shapes. As shown in Fig. 1, intermetallic compound phases containing the element M and at least one of Fe and Co

have a lateral size (s) of 0.8  $\mu\text{m}$  or less, and the closest portions of the intermetallic compound phases containing the element M and at least one of Fe and Co are located at a distance d of 0.6  $\mu\text{m}$  or less. The phase between the intermetallic compound phases containing the element M is based on Fe and Co.

5 [0022]

The Fe-Co-M alloy sputtering target material of the present invention makes it possible to deposit a desirable thin soft magnetic layer by sputtering without cracking of the sputtering target material during the sputtering. Thus, a desirable perpendicular magnetic recording medium including the thin soft magnetic layer of the present invention can be provided.

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## EXAMPLES

[0023]

The present invention will now be described in more detail with reference to the following examples.

15 [0024]

Soft magnetic alloy powders having the compositions shown in Table 1 were prepared by gas atomization. The resulting powders were classified into 500  $\mu\text{m}$  or less for use as powder feedstocks for consolidation by hot isostatic pressing (HIP). Billets for HIP were prepared by placing the powder feedstocks into carbon steel cans with a diameter of 250  
20 mm and a length of 50 mm, capping the cans, degassing the cans in a vacuum, and sealing the vent. The billets filled with the powder were consolidated by HIP at the consolidation pressures and consolidation temperatures for the retention times shown in Table 1. Sputtering target materials having a diameter of 180 mm and a thickness of 7 mm were then prepared from the consolidated bodies. Eight sputtering target materials  
25 were sputtered. After sputtering was continued until the thickness of the thinnest portion reached 1.5 mm, the number of cracked targets in the eight targets was counted. An example where no or one target cracked was determined to be acceptable.

[0025]

[Table 1]

| No. | Composition of alloy (at%) |    |    |    |    |    |    | Area of eutectic structure (%) | Cracking during sputtering (number of cracked targets among 8 targets) | Consolidation pressure (MPa) | Consolidation temperature (°C) | Retention time (hr) | Remarks           |
|-----|----------------------------|----|----|----|----|----|----|--------------------------------|------------------------------------------------------------------------|------------------------------|--------------------------------|---------------------|-------------------|
|     | Fe                         | Co | Ta | Nb | Mo | W  | Cr |                                |                                                                        |                              |                                |                     |                   |
| 1   | 53                         | 29 | 18 | 0  | 0  | 0  | 0  | 16                             | 0                                                                      | 130                          | 970                            | 2                   | Inventive example |
| 2   | 43                         | 29 | 28 | 0  | 0  | 0  | 0  | 27                             | 1                                                                      | 130                          | 970                            | 2                   |                   |
| 3   | 46                         | 29 | 25 | 0  | 0  | 0  | 0  | 19                             | 0                                                                      | 130                          | 970                            | 2                   |                   |
| 4   | 51                         | 29 | 20 | 0  | 0  | 0  | 0  | 18                             | 0                                                                      | 130                          | 970                            | 2                   |                   |
| 5   | 59                         | 29 | 12 | 0  | 0  | 0  | 0  | 5                              | 0                                                                      | 130                          | 970                            | 2                   |                   |
| 6   | 63                         | 29 | 8  | 0  | 0  | 0  | 0  | 3                              | 0                                                                      | 150                          | 1200                           | 2                   |                   |
| 7   | 67                         | 29 | 4  | 0  | 0  | 0  | 0  | 1                              | 0                                                                      | 150                          | 1200                           | 2                   |                   |
| 8   | 7                          | 65 | 0  | 28 | 0  | 0  | 0  | 23                             | 0                                                                      | 130                          | 1100                           | 2                   |                   |
| 9   | 10                         | 65 | 0  | 25 | 0  | 0  | 0  | 22                             | 1                                                                      | 130                          | 1100                           | 2                   |                   |
| 10  | 15                         | 65 | 0  | 20 | 0  | 0  | 0  | 20                             | 0                                                                      | 130                          | 1100                           | 2                   |                   |
| 11  | 17                         | 65 | 0  | 18 | 0  | 0  | 0  | 15                             | 0                                                                      | 130                          | 1000                           | 2                   |                   |
| 12  | 25                         | 65 | 0  | 10 | 0  | 0  | 0  | 12                             | 0                                                                      | 130                          | 1000                           | 2                   |                   |
| 13  | 27                         | 65 | 0  | 8  | 0  | 0  | 0  | 10                             | 0                                                                      | 130                          | 1000                           | 2                   |                   |
| 14  | 31                         | 65 | 0  | 4  | 0  | 0  | 0  | 5                              | 0                                                                      | 130                          | 1150                           | 2                   |                   |
| 15  | 42                         | 30 | 0  | 0  | 28 | 0  | 0  | 25                             | 1                                                                      | 130                          | 1100                           | 2                   |                   |
| 16  | 52                         | 30 | 0  | 0  | 18 | 0  | 0  | 18                             | 1                                                                      | 130                          | 1100                           | 2                   |                   |
| 17  | 62                         | 30 | 0  | 0  | 8  | 0  | 0  | 4                              | 0                                                                      | 130                          | 1100                           | 2                   |                   |
| 18  | 7                          | 65 | 0  | 0  | 0  | 28 | 0  | 22                             | 1                                                                      | 130                          | 1000                           | 2                   |                   |
| 19  | 17                         | 65 | 0  | 0  | 0  | 18 | 0  | 12                             | 0                                                                      | 130                          | 1000                           | 2                   |                   |
| 20  | 27                         | 65 | 0  | 0  | 0  | 8  | 0  | 6                              | 0                                                                      | 130                          | 1000                           | 2                   |                   |
| 21  | 42                         | 30 | 0  | 0  | 0  | 0  | 28 | 18                             | 0                                                                      | 130                          | 1100                           | 2                   |                   |
| 22  | 52                         | 30 | 0  | 0  | 0  | 0  | 18 | 10                             | 0                                                                      | 130                          | 1100                           | 2                   |                   |
| 23  | 62                         | 30 | 0  | 0  | 0  | 0  | 8  | 3                              | 0                                                                      | 130                          | 1100                           | 2                   |                   |
| 24  | 7                          | 65 | 0  | 0  | 0  | 0  | 0  | 17                             | 0                                                                      | 130                          | 1000                           | 2                   |                   |
| 25  | 17                         | 65 | 0  | 0  | 0  | 0  | 0  | 9                              | 0                                                                      | 130                          | 1000                           | 2                   |                   |

[0026]

[Table 2]

| No. | Composition of alloy (at%) |    |    |    |    |   | Area of eutectic structure (%) | Cracking during sputtering (number of cracked targets among 8 targets) | Consolidation pressure (MPa) | Consolidation temperature (°C) | Retention time (hr) | Remarks           |
|-----|----------------------------|----|----|----|----|---|--------------------------------|------------------------------------------------------------------------|------------------------------|--------------------------------|---------------------|-------------------|
|     | Fe                         | Co | Ta | Nb | Mo | W | Cr                             | V                                                                      |                              |                                |                     |                   |
| 26  | 27                         | 65 | 0  | 0  | 0  | 0 | 0                              | 8                                                                      | 130                          | 1000                           | 2                   | Inventive example |
| 27  | 73                         | 0  | 15 | 12 | 0  | 0 | 0                              | 0                                                                      | 120                          | 1150                           | 2                   |                   |
| 28  | 73                         | 0  | 12 | 15 | 0  | 0 | 0                              | 0                                                                      | 130                          | 1150                           | 2                   |                   |
| 29  | 74                         | 0  | 13 | 13 | 0  | 0 | 0                              | 0                                                                      | 150                          | 1150                           | 2                   |                   |
| 30  | 52                         | 30 | 9  | 9  | 0  | 0 | 0                              | 0                                                                      | 150                          | 1000                           | 2                   |                   |
| 31  | 52                         | 30 | 9  | 0  | 9  | 0 | 0                              | 0                                                                      | 150                          | 1000                           | 2                   |                   |
| 32  | 52                         | 30 | 9  | 0  | 0  | 9 | 0                              | 0                                                                      | 150                          | 1200                           | 2                   |                   |
| 33  | 52                         | 30 | 9  | 0  | 0  | 0 | 9                              | 0                                                                      | 150                          | 1000                           | 2                   |                   |
| 34  | 52                         | 30 | 9  | 0  | 0  | 0 | 0                              | 9                                                                      | 150                          | 1000                           | 2                   |                   |
| 35  | 52                         | 30 | 0  | 9  | 9  | 0 | 0                              | 0                                                                      | 150                          | 1000                           | 2                   |                   |
| 36  | 52                         | 30 | 0  | 9  | 0  | 9 | 0                              | 0                                                                      | 150                          | 1200                           | 2                   |                   |
| 37  | 0                          | 82 | 0  | 9  | 0  | 0 | 0                              | 0                                                                      | 150                          | 1000                           | 2                   |                   |
| 38  | 0                          | 82 | 0  | 9  | 0  | 0 | 0                              | 9                                                                      | 150                          | 1000                           | 2                   |                   |
| 39  | 17                         | 65 | 0  | 0  | 9  | 9 | 0                              | 0                                                                      | 150                          | 1050                           | 2                   |                   |
| 40  | 52                         | 30 | 0  | 0  | 9  | 0 | 9                              | 0                                                                      | 150                          | 1000                           | 2                   |                   |
| 41  | 17                         | 65 | 0  | 0  | 9  | 0 | 0                              | 9                                                                      | 150                          | 1000                           | 2                   |                   |
| 42  | 32                         | 50 | 0  | 0  | 0  | 9 | 9                              | 0                                                                      | 150                          | 1000                           | 2                   |                   |
| 43  | 32                         | 50 | 0  | 0  | 0  | 9 | 0                              | 9                                                                      | 150                          | 1000                           | 2                   |                   |
| 44  | 37                         | 45 | 0  | 0  | 0  | 0 | 9                              | 9                                                                      | 150                          | 1000                           | 2                   |                   |
| 45  | 62                         | 30 | 4  | 4  | 0  | 0 | 0                              | 0                                                                      | 120                          | 1000                           | 2                   |                   |
| 46  | 53                         | 29 | 18 | 0  | 0  | 0 | 0                              | 0                                                                      | 150                          | 1000                           | 2                   |                   |
| 47  | 53                         | 29 | 18 | 0  | 0  | 0 | 0                              | 0                                                                      | 130                          | 970                            | 2                   |                   |
| 48  | 53                         | 29 | 18 | 0  | 0  | 0 | 0                              | 0                                                                      | 150                          | 970                            | 2                   |                   |

[0027]

[Table 3]

| No. | Composition of alloy (at%) |      |     |     |     |     |     | Area of eutectic structure (%) | Cracking during sputtering (number of cracked targets among 8 targets) | Consolidation pressure (MPa) | Consolidation temperature (°C) | Retention time (hr) | Remarks             |
|-----|----------------------------|------|-----|-----|-----|-----|-----|--------------------------------|------------------------------------------------------------------------|------------------------------|--------------------------------|---------------------|---------------------|
|     | Fe                         | Co   | Ta  | Nb  | Mo  | W   | Cr  |                                |                                                                        |                              |                                |                     |                     |
| 49  | 57.2                       | 30.8 | 12  | 0   | 0   | 0   | 0   | 40                             | 7                                                                      | 120                          | 950                            | 1                   | Comparative example |
| 50  | 46                         | 40   | 14  | 0   | 0   | 0   | 0   | 35                             | 8                                                                      | 120                          | 950                            | 1                   |                     |
| 51  | 42                         | 29   | 29  | 0   | 0   | 0   | 0   | 16                             | 8                                                                      | 150                          | 1200                           | 2                   |                     |
| 52  | 6                          | 65   | 0   | 29  | 0   | 0   | 0   | 16                             | 8                                                                      | 150                          | 1200                           | 2                   |                     |
| 53  | 42                         | 29   | 0   | 0   | 29  | 0   | 0   | 18                             | 8                                                                      | 150                          | 1200                           | 2                   |                     |
| 54  | 6                          | 65   | 0   | 0   | 0   | 29  | 0   | 20                             | 8                                                                      | 150                          | 1200                           | 2                   |                     |
| 55  | 42                         | 29   | 0   | 0   | 0   | 0   | 29  | 28                             | 8                                                                      | 150                          | 1200                           | 2                   |                     |
| 56  | 6                          | 65   | 0   | 0   | 0   | 0   | 0   | 28                             | 8                                                                      | 150                          | 1200                           | 2                   |                     |
| 57  | 32                         | 65   | 3   | 0   | 0   | 0   | 0   | 35                             | 0                                                                      | 120                          | 950                            | 1                   |                     |
| 58  | 32                         | 65   | 0   | 3   | 0   | 0   | 0   | 35                             | 0                                                                      | 120                          | 950                            | 1                   |                     |
| 59  | 32                         | 65   | 0   | 0   | 3   | 0   | 0   | 34                             | 0                                                                      | 120                          | 950                            | 1                   |                     |
| 60  | 32                         | 65   | 0   | 0   | 0   | 3   | 0   | 34                             | 0                                                                      | 120                          | 950                            | 1                   |                     |
| 61  | 32                         | 65   | 0   | 0   | 0   | 0   | 3   | 50                             | 0                                                                      | 120                          | 950                            | 1                   |                     |
| 62  | 32                         | 65   | 0   | 0   | 0   | 0   | 0   | 50                             | 0                                                                      | 120                          | 950                            | 1                   |                     |
| 63  | 32                         | 65   | 1.5 | 1.5 | 0   | 0   | 0   | 35                             | 0                                                                      | 120                          | 950                            | 1                   |                     |
| 64  | 32                         | 65   | 1.5 | 0   | 1.5 | 0   | 0   | 33                             | 0                                                                      | 120                          | 950                            | 1                   |                     |
| 65  | 32                         | 65   | 1.5 | 0   | 0   | 1.5 | 0   | 34                             | 0                                                                      | 120                          | 950                            | 1                   |                     |
| 66  | 32                         | 65   | 1.5 | 0   | 0   | 0   | 1.5 | 45                             | 0                                                                      | 120                          | 950                            | 1                   |                     |
| 67  | 32                         | 65   | 1.5 | 0   | 0   | 0   | 0   | 50                             | 0                                                                      | 120                          | 950                            | 1                   |                     |

Nos. 1 to 48 shown in Tables 1 and 2 are inventive examples, and Nos. 49 to 67 shown in Table 3 are comparative examples.

[0028]

- 5 Fig. 2 is an electron micrograph showing a reflected electron image of No. 1 in Table 1. As shown in Fig. 2, in which the reflected electron image shows a contrast depending on the atomic number, the sputtering target material of No. 1 has a structure including intermetallic compound phases containing the element M, which are gray, and Co-Fe phases, which are dark gray. The results of image analysis show S1 (area of eutectic structure) / S2 (area of examination field of view)  $\times$  100 (%) = 15%.
- 10

[0029]

- In Comparative Example No. 49 shown in Table 3, which has an area (S1) ratio of the eutectic structure of 40%, the sputtering target materials crack during sputtering. In Comparative Example No. 50, which has an area (S1) ratio of the eutectic structure of 35%, the sputtering target materials crack during sputtering. In Comparative Example Nos. 51 to 56, which have an area (S1) ratio of the eutectic structure of 16% to 28% and an element M content of 29 at.%, the sputtering target materials crack during sputtering.
- 15

[0030]

- In Comparative Example Nos. 57 to 62, which have a high area (S1) ratio of the eutectic structure of 34% to 50% and a low element M content of 3 at.%, the sputtering target materials do not crack during sputtering. In Comparative Example Nos. 63 to 67, which have a high area (S1) ratio of the eutectic structure of 33% to 50% and a low total element M content of 3 at.%, the sputtering target materials do not crack during sputtering. In contrast, the inventive examples shown in Tables 1 and 2, all of which satisfy the conditions according to the present invention, provide a Fe-Co alloy sputtering target material that does not crack during sputtering.
- 20
- 25

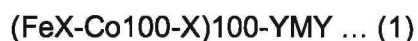
[0031]

As described above, fractures, which are prevented from propagating by the FeCo phase,

readily propagate through the compound phases in the eutectic structure, thus leading to the cracking of the target. Accordingly, the microstructure of the sputtering target material is controlled to provide a Fe-Co alloy sputtering target material that does not crack during sputtering.

## CLAIMS

1. A Fe-Co alloy sputtering target material comprising a Fe-Co-M alloy represented by formula (1):



wherein the atomic ratios are  $0 \leq X \leq 100$  and  $15 \leq Y \leq 28$ , and the element M includes at least one element selected from Nb, Ta, Mo, W, Cr, and V,

the sputtering target material having a microstructure comprising a phase based on Fe and Co and an intermetallic compound phase comprising the element M and at least one of Fe and Co, the area ratio of a eutectic structure in the overall microstructure being 30% or less.

2. A method for manufacturing the Fe-Co-M alloy sputtering target material according to Claim 1, the method comprising the step of consolidating an alloy powder by hot isostatic pressing (HIP) at a consolidation temperature of 970 to 1200°C and a consolidation pressure of 130 to 150 MPa, wherein the alloy powder is prepared by gas atomization of a melt of the alloy.