The method taught herein can produce a highly cross-linked polypropylene material, for instance in the form of a layer, having a relatively low dielectric constant. Moreover, the three dimensionally cross-links formed in the polypropylene ensure that the material or layer is relatively thermally stable, and further that it exhibits mechanical properties after Ashby, consistent with ceramics. PECVD production of the layer does not rely on solvents or water. The resulting consistency, thermal stability and low dielectric constant of the layer produced by the taught method make it well suited to use as a dielectric layer in the manufacture of integrated circuits. Advantageously, the present invention provides a single process step to create both polypropylene polymer chains and cross-links between them, and does not require an additional curing step in order to provide these cross-links.

At lower pressures, the cross-linked polypropylene can be formed as a continuous layer on a substrate. According to preferred methods, the pressure is selected to be less than 5 Torr (0.667 kPa) in order to produce a continuous layer on the substrate where this is desired. In other preferred methods, particularly where the cross-linked polypropylene is desired as flakes or nano-particles formed in the plasma phase, the pressure is selected to be greater than 5 Torr (0.667 kPa).

The mechanical stress in the polypropylene layer is typically inversely proportional to pressure, due to the greater energy of the ion bombardment on the substrate. Ion bombardment is an intrinsic part of the plasma formation process that can be controlled by the use of the power coupled into the plasma, the pressure and the electrode configurations among other considerations. Those skilled in the art could perform the ion bombardment via other processes. Amongst other things, this ion bombardment affects the adhesion of the layer to the substrate and the surface energies. In preferred embodiments, therefore, the pressure within the chamber is selected to be greater than 200 mTorr (0.0267 kPa).

The mechanical stress in the cross-linked polypropylene layer is also a function of the power per unit area applied to the plasma electrode. The greater the applied power, the greater the rate of growth of the cross-linked polypropylene layer, but also the greater the mechanical stress in the layer. As such, in preferred embodiments, the applied power per unit area of the plasma electrode is less than

substrate 4 is mounted. The substrate 4 is, in this embodiment, formed of silicon. However, other materials may be used as a substrate. For example, semiconducting materials, such as germanium, may be used. Alternatively, metals may also be used.

At the top of the chamber 2 is a showerhead 5, which functions as a gas inlet and plasma electrode. More specifically, the showerhead 5 has an inlet 6 though which it receives feedstock gas for use in the PECVD process and a plurality of outlets 7 through which the feedstock gas can pass out of the showerhead 5 and into the chamber 2. The showerhead 5 is preferably metallic. Although the showerhead 5 functions as an electrode in this embodiment, additional or alternative electrode structures may be used.

A power supply 8 is provided that can apply a voltage to the showerhead 5. In preferred embodiments, the power supply 8 provides an alternating current (AC) at a frequency of around 13.56 MHz. Other frequencies may be used, although they are preferably at least 1Hz. However, in other embodiments the power supply 8 may provide AC at different frequencies or may apply a direct current (DC). Nevertheless, AC is preferred because it negates the risk of charge build up at the electrodes and therefore allows the plasma to be struck at lower power levels. Switched power or linearly controlled bipolar power may be coupled to the plasma to dissociate the gases and minimise ion bombardment. The power provided by the power supply 8 is limited to avoid damage to the deposited layer that would otherwise be caused by ion bombardment.

At the bottom of the chamber 2 is a gas outlet 9 through which gas in the chamber 2 can be evacuated using a vacuum pump 10. In this embodiment, the vacuum pump 10 is a turbo molecular pump. In another embodiment, the vacuum pump 10 is a rotary pump. The vacuum pump 10 is capable of reducing the pressure in the chamber 2 to as low as around 5e-7 Torr (6.67e-11 kPa).

An acetylene (C_2H_2) supply vessel 11 is also provided. Alternative carbon containing gases to acetylene may also be used. The acetylene supply vessel 11 provides acetylene gas into the chamber at a rate controlled by a mass flow controller 12. A filter 13 may be included to filter the supply of acetylene from the acetylene supply vessel 11. A supplementary gas supply vessel 14 is also

using an adjustable valve between the chamber and the vacuum pump, or by regulating the flow rate of the gases. In a preferred embodiment, the pressure is regulated to be greater than 200mTorr(0.0267kPa). At lower pressures, the energy of ion bombardment on the substrate 4 is higher and may cause damage to the polypropylene layer and, in particular operating conditions further cause plasma instability.

Once the feedstock gas is in the chamber 2, the power supply 8 provides an AC or a DC to the showerhead 5 in order to strike a plasma in the chamber 2. The plasma is then maintained in a steady state and the process of PECVD occurs. As a result, the highly cross-linked polypropylene film is deposited on the substrate. It is possible to provide a heater (not shown) to apply additional heat to the substrate to increase the thermal stability of the cross-linked polypropylene film. In preferred embodiments, the heater is used to apply heat at a temperature of between 100°C to 1000°C, more preferably between 200°C to 500°C, and most preferably between 250°C and 300°C. UV plasma bombardment during this process may be used.

The mechanism by which the cross-linked polypropylene forms, differs according to the pressure in the chamber 2. At pressures above approximately 5 Torr (0.667kPa) depending upon specific operating conditions, highly cross-linked polypropylene is produced within the plasma and is then deposited on the substrate. At pressures below approximately 5 Torr, the highly cross-linked polypropylene is produced directly on the substrate 4 itself. The difference between these two processes affects the properties of the cross-linked polypropylene film or material.

Above approximately 5 Torr (0.667kPa) the highly cross-linked polypropylene nucleates in the plasma phase, and comprises a plurality of distinct particles that settle together to form the layer on the substrate 4. As a result, there are regions in the layer that are left empty, taking on whatever atmosphere the layer is placed in. This has a beneficial effect in terms of the effective k-value, as the k-value of air is very low (approximately 1). However, the material nucleated within the plasma phase does not provide a smooth upper surface to facilitate bonding of additional layers. Where necessary, post processing can palanarise the layer to create very

smooth surfaces for integration to device structures, or the mixing with suitable epoxies may allow for thin films to be produced.

At pressures below approximately 5 Torr (0.667 kPa), the cross-linked material nucleates directly on the substrate 4. Its physical properties are different, particularly as it forms a continuous layer on the substrate 4 with a smooth surface.

Figures 2A and 2B show the spectra 201, 204 of the material nucleated in the plasma phase (henceforth "Material A") and on the substrate (henceforth "Material B") obtained from a Fourier transform infra-red (FTIR) spectroscopy apparatus. The spectrum 202 of a control sample of conventionally produced polypropylene is also shown.

It can be seen from Figures 2A and 2B that Material A 201 produced at pressures above 5 Torr and Material B 204 deposited at pressures below 5 Torr (0.667 kPa) share a number of absorption peaks with the control sample of polypropylene 202. It can be surmised from this that both Materials A and B have a polypropylene-like backbone structures (that is, they include polypropylene polymer chains). The additional peaks of the spectra 201, 204 of Materials A and B show, however, that they differ from standard polypropylene 202. In particular, the spectra 201, 204 of Materials A and B both show a peak associated with a C=C double bond (an oleophinic bond). This bond is associated with the cross-linking of polymer chains, with increased cross-linking having the macroscopic effect of enhancing the temperature stability of the material, and also providing certain mechanical advantages such as low creep and enhanced mechanical integrity.

The energy within the plasma assists in the production of cross-links between the polymer chains. This energy typically includes ultraviolet radiation, although it may be released in other forms. The use of an ultraviolet radiation containing plasma, for example, can effectively provide a combined singular polymer production and curing process step, assisting in the direct production of a cross-linked polypropylene layer with excellent macroscopic properties. The plasma has an ultraviolet component, and preferably also has higher energy plasma species, ions and electrons.

CLAIMS

1. A method of producing a highly cross-linked polymer material including the steps of:

providing a reaction chamber;

selecting one or more carbon containing gases from a plurality of carbon containing gases, wherein at least one of the gases is acetylene;

feeding said one or more selected carbon containing gases into said chamber;

feeding acetone into said chamber;

feeding a carrier gas which includes hydrogen into said chamber;

wherein the pressure in said chamber is set to be greater than 200 mTorr (0.0267 kPa) and less than 5 Torr (0.667 kPa);

striking a plasma in said chamber, said plasma causing said gases to dissociate into a phase including methyl radicals;

causing said dissociated phase to nucleate and thereby to create highly cross-linked polymer material.

2. A method as claimed in claim 1, comprising the step of annealing the cross linked polymer material in a vacuum or a controlled gas environment, wherein the controlled gas environment uses one or a composition of inert gases.

3. A method according to claim 2, wherein said annealing step is performed so as to change or reduce the dielectric constant of said nucleated polymer material.

4. A method as claimed in claim 2 or 3, wherein annealing is performed at a temperature greater than 100°C.

5. A method as claimed in any of claims 2 to 4, wherein said annealing step is carried out for a period of at least ten minutes.