Thin films of tantalum were obtained by sputtering tantalum onto glass in a vacuum of 10^{-6} Torr. The thickness of the film was about 500 Å, with area 25 mm x 25 mm. To obtain implanted material, tantalum films of 500 Å thickness were implanted with nitrogen ions of energy 40 keV and a dose of 1.6×10^4 ions cm^{-2}. Strips 1 mm wide were cut from unimplanted and nitrogen implanted tantalum films. They were mounted in Philips powder cameras, 11.46 cm diameter. Cu$K\alpha$ radiation with a nickel filter was used. The specimens were not rotated. The specimens were then annealed in evacuated quartz tubes at 400, 500 and 600° C for 1 h. X-ray powder photographs were taken after each anneal.

The *d* spacings of the reflection lines from the unannealed specimens corresponded to those of b c c tantalum and β-tantalum. Both phases showed preferred orientation. After annealing at temperatures of 400 and 500° C, the X-ray diffraction patterns showed little change from those of the as-sputtered tantalum. Annealing at 600° C caused the rings from b c c tantalum and β-tantalum to disappear. The diffraction pattern of the implanted unannealed specimen showed some continous rings from TaN. Preferred orientation of the β-tantalum was observed but not of the b c c tantalum. Annealing at 600° C caused the TaN rings to sharpen, indicating that it had recrystallized.

The dislocation density for b c c crystals can be calculated from the equation given by Williamson and Smallman [4] as follows:

$$\rho = 14.4 \frac{E^2}{b^2}$$

where ρ = dislocation density; E = strain value; b = Burgers vector.

It can be seen from Fig. 1 that the microstrain dropped by about 25% on heating from room temperature to 600° C, thus the dislocation density, which varies as the $(\text{strain})^2$, will drop by over 40% with a corresponding effect on the resistivity.

The formation of the β-tantalum in the sputtered tantalum films is in agreement with the observations of Read and Altman [5]. They found that β-tantalum is unstable, changing into b c c tantalum at ~700° C when heated in a vacuum of 10^{-8} Torr.

It can thus be seen that heat produces many complex structural changes in ion-implanted glass supported tantalum films. It may be assumed that most of these changes will produce corresponding changes in the electrical properties of the films.

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A relationship between undercooling and atomized powder diameter

An expression between undercooling ΔT, and atomized powder diameter D can be derived if we consider that when continuous cooling occurs the undercooling will be [1]

$$\Delta T^2 = k(\dot{T}) \qquad (1)$$

in which k is a constant depending on the system being solidified and $\dot{T}$ is the cooling rate. The above equation should be valid for any composition invariant transformation in which the nucleation of the product phase can be described by classical nucleation theory [1]. Equation 1 applies to nucleation during continuous cooling and we apply it here to undercooled atomized particles experiencing a high cooling rate, assuming that the solidification process immediately after nucleation is composition invariant. Assuming that a sphere contains negligible temperature gradients, the cooling rate $\dot{T}$ for convection and radiation cooling will be given by

$$\dot{T} = \frac{6}{D\rho C_p}[h_c(T-T) + \epsilon\sigma(T^4 - T_0^4)]. \qquad (2)$$

In Equation 2 D and ρC_p are the diameter and the heat capacity per unit volume of the atomized droplet, ϵ, σ and h_c are the emissivity of the powder, the Stephan–Boltzmann constant and the convection heat transfer coefficient, respectively, while T_0 is the temperature of the surrounding medium. Since one expects that the powder diameter should be inversely proportional to its velocity, the convection coefficient h_c for a sphere moving through a gas under turbulent flow for all atomization processes can be expressed as [2]

$$h_c = \lambda/D^m. \qquad (3)$$

In Equation 3 m could be obtained theoretically or from the experimental data while λ is proportional to P, where P is the gas pressure and should be approximately constant for a given process and material.

Combining Equations 1 to 3 yields the final result

$$\Delta T^2 = K\left(\frac{6}{D\rho C_p}\right)\left[\frac{\lambda}{D^m}(T-T_0) + \epsilon\sigma(T^4 - T_0)\right] \qquad (4)$$

The values [2] of $T = 1773\,\text{K}$, $\epsilon = 0.33$, $\rho C_p = 1.27\,\text{cal cm}^{-3}\,\text{K}^{-1}$, and $\lambda = 0.5 \times 10^{-3}\,\text{cal cm}^{-1}\,\text{sec}^{-1}\,\text{K}^{-1}$ for maraging 300 alloy [4] can be used in Equation 5, while K can be determined theoretically or experimentally.

Equation 4 estimates the amount of undercooling, assuming it is not very high, if the powder size and the rest of the parameters are known. Of interest in Equation 5 is the prediction that when radiation cooling dominates we will have that

$$D \cdot \Delta T^2 = \text{const.}, \qquad (5)$$

while for convection cooling we should have

$$D^{m+1}\Delta T^2 = \text{const.} \qquad (6)$$

A rough guess for the rotating electrode process [2] is that $m = 1$, so Equation 7 becomes

$$D \cdot \Delta T = \text{const.} \qquad (7)$$

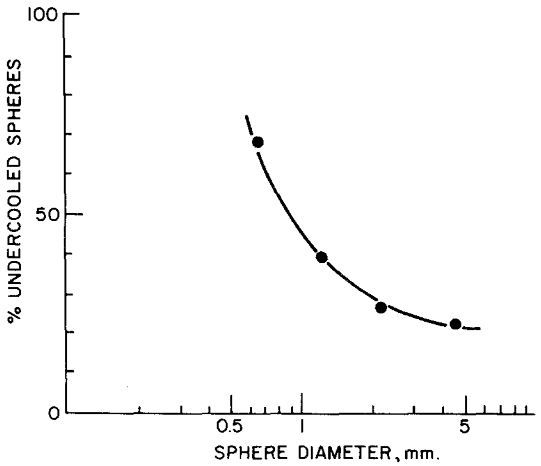

Figure 1 Percentage (%) of undercooled spheres versus diameter for maraging 300 alloy atomized in argon.

If a process were modified to decrease λ, for example by reducing the gas pressure in half, the undercooling will be reduced by 1.2.

Equations 5 and 6 show that for smaller diameter particles the undercooling increases while for larger diameters it decreases. Recent studies [3] for maraging 300 alloy atomized in argon (see Fig. 1) shows a similar trend between the percentage (%) of undercooled spheres and sphere diameter.

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