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VOID-STRENGTHENING IN ALUMINUM AND ITS NATURE

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Abstract—Temperature and strain rate dependence of yield strength were used to analyze the nature of aluminum strengthened by the formation of voids. Aluminum rods 99.999% pure were quenched and heat treated to form voids with an approximate density of 10^{13}–10^{14} voids/cm^3. Voids in selected samples were observed by electron microscopy. The yield strength of the void strengthened samples was measured at various temperatures from 77 to 593 K and at two strain rates, 3.33×10^{-2}/s and 1.67×10^{-3}/s. Tests at room temperature and at 77 K were made at various strain rates.

The similarity of Coulomb's approach to void strengthening and that of Orowan stress with Ashby's dipole criterion was observed, and it was determined that the extent and effectiveness of void strengthening depends primarily on void density and much less on void size. The amount of strengthening obtained at room temperature was found to be consistent with the estimates. Compared to annealed aluminum, void strengthened aluminum is more susceptible to the instability of plastic flow at low temperatures; its temperature dependence of yield strength varies in different temperature ranges and shows a higher strain rate sensitivity of yield strength.

Résumé—On a analysé la nature du durcissement de l'aluminium par formation de cavités, en étudiant la variation de la limite élastique en fonction de la température et de la vitesse de déformation. On a trempé et traité thermiquement des barres d'aluminium 99,999% pour former des cavités dont la densité allait approximativement de 10^{13} à 10^{14} cavités/cm^3. On a observé en microscopie électronique des cavités dans quelques échantillons choisis. On a mesuré la limite élastique des échantillons durcis par les cavités à diverses températures comprises entre 77 et 593 K et pour des vitesses de déformation égales à $3{,}33 \times 10^{-2}$ et $1{,}67 \times 10^{-3}$ sec^{-1}. On a effectué des essais à l'ambiante et à 77 K pour diverses vitesses de déformation.

On a observé la ressemblance entre l'approche de Coulomb du durcissement par cavités et celle de la contrainte d'Orowan avec le critère du dipôle d'Ashby, et l'on a déterminé que l'importance et l'efficacité du durcissement par cavités dépend essentiellement de la densité des cavités et beaucoup moins de leur taille. Le taux de durcissement à l'ambiante était en bon accord avec les valeurs estimées. L'aluminium durci par cavités est plus sujet que l'aluminium recuit à une instabilité de la déformation plastique à basse température; la variation de la limite élastique en fonction de la température dépend du domain de température; la limite élastique est plus sensible à la vitesse de déformation.

Zusammenfassung—Aus der Abhangigkeit der Fließspannung von Temperatur und Dehngeschwindigkeit wurde auf die Verfestigung von Aluminium durch Ausbildung von Hohlräumen geschlossen. Aluminiumstäbe mit einer Reinheit von 99,999% wurden zur Erzeugung von Hohlräumen in einer Dichte von etwa 10^{13} bis 10^{14} cm^{-3} abgeschreckt und wärmebehandelt. Die Hohlräume wurden elektronenmikroskopisch in ausgewählten Proben untersucht. Die Fließspannung der hohlraumverfestigten Proben wurde bei verschiedenen Temperaturen zwischen 77 und 593 K und bei zwei Dehngeschwindigkeiten ($3{,}33 \times 10^{-2}$ und $1{,}67 \times 10^{-3}$ sec^{-1}) gemessen. Bei Raumtemperatur und 77 K wurden Versuche mit mehreren Dehngeschwindigkeiten durchgeführt.

Unter Berücksichtigung der Ähnlichkeit von Coulombs Näherung mit der Hohlraumverfestigung und derjenigen der Orowanspannung mit Ashbys Dipolkriterium wurde bestimmt, daß Ausmaß und Wirksamkeit der Hohlraumverfestigung in erster Linie von der Hohlraumdichte abhängt, weit weniger von der Hohlraumgröße. Die Höhe der Raumtemperaturverfestigung stimmt mit den Abschätzungen überein. Im Vergleich zu geglühtem Aluminium ist hohlraumverfestigtes Aluminium bei tiefen Temperaturen empfindlicher gegenüber Fließinstabilitäten; die Temperaturabhängigkeit der Fließspannung von hohlraumverfestigtem Aluminium ist in verschiedenen Temperaturbereichen unterschiedlich, die Fließspannung zeigt eine stärkere Abhängigkeit von der Dehngeschwindigkeit.

INTRODUCTION

Makin *et al.* [1] observed a consistent relationship between the increased critical shear stress in neutron irradiated copper crystals and the density of black dot defects (vacancy clusters that are below the 50 Å size and considered to be different from dislocation loops). However, they found that a similar and consistent relationship does not exist when the strength is related to larger vacancy clusters, i.e. the dislocation loops present in the samples. Similarly, Westmacott [2] found that the value of the increased shear stress is not related to the size and distribution of the observed dislocation loops in the quenched aluminum nor is it related during the process when the loops are annealed out. These two studies clearly show that the presence of the different types of vacancy clusters other than the observable dislocation loops cause a marked strengthening in neutron irradiated copper and in quenched aluminum. Hardie and Michael [3] found the same to be true in their experiments on aging quenched specimens of aluminum and Al–Mg alloys.

The available theories, i.e. void-strengthening by the Coulomb [4] theory and strengthening caused by tetragonal distortions by the Fleischer [5, 6] theory, fail to distinguish between the temperature dependence of the yield strength for voids and for loops. The deductions that have been drawn from these theories concerning the temperature dependence of yield strength are incorrect only because the models were inadequate in this respect. Therefore, Westmacott's [2] inference, which is based on Fleischer's theory, that the strengthening in quenched aluminum samples is caused by clusters of vacancies thought to be ungrown loop nuclei that consist of 6–7 vacancies in a collapsed configuration has to be accepted with reservations. His observation that the larger loops do not cause increased shear stress is more striking. Loop nucleation and void nucleation are rapid processes that occur within a few seconds after quenching, consequently the question arises as to why it takes a much longer period of time (a few hours) after the quenching for the greater strength to develop. This indicates that either those vacancy clusters responsible for the strengthening become somewhat larger than the ungrown loop nuclei or it is necessary for the vacancies to form certain other vacancy clusters to be effective obstacles to the dislocation motion.

EXPERIMENTAL PROCEDURE

Aluminum rod samples 3.1 mm dia. were prepared by rolling and swaging 99.999% aluminum that was supplied by the United Mineral and Chemical Corporation, New York. Although care was taken to keep from contaminating the material, which had initial impurities of Cu (2 ppm), Fe (5 ppm), Mg (0.1 ppm), and Si (1 ppm), the final analysis showed a sharp rise in iron content to about 30 ppm.

During the heat treatment for void formation by quenching and aging, it was found, after quenching, that some of the specimens crack longitudinally. To sort out defective samples, all were first heated to 600°C and then quenched in water at room temperature. After repeating this process three times, the cracked samples were rejected.

The quenching and aging treatment [7–9] developed for producing voids in aluminum was successfully adapted with some modifications as shown schematically in Fig. 1. The specimens were placed in the longitudinal groove of a stainless steel core, which was heated in a tube furnace to 640°C for 2 h. The hot core containing the sample was drawn out and quickly rotated so that the specimen dropped into a 45% Ucon aqueous solution at 20°C as suggested by Westmacott [10]. The sample was quickly removed in a wire mesh tray, washed in water at

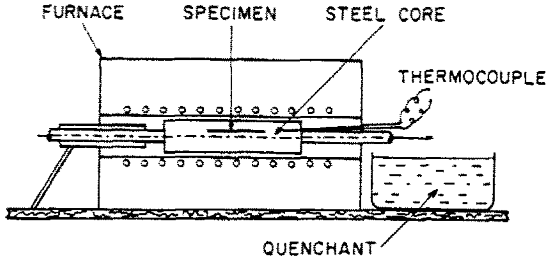

Fig. 1. Experimental furnace and quenching apparatus.

90–100°C, and transferred to an oven at 105 ± 2°C to age for about 5 h. The time required for moving the samples from the initial quench to the oven was about 15 s.

Voids in some of the samples were observed by transmission electron microscopy after the specimens had been cut transversely with a wire saw to yield discs 0.4 mm thick. The discs were electropolished by a single stage, twin jet process described by Hacking *et al.* [11].

The yield strength of the samples was measured at various temperatures between 77 and 593°K and at strain rates 3.33×10^{-2}/s and 1.67×10^{-3}/s. Tensile test at room temperature and at 77 K in liquid nitrogen were carried out at various strain rates between 3.33×10^{-5}/s and 3.33×10^{-2}/s. The gage length was maintained at 1 in., and the yield stress was taken as 0.1% proof stress. Each test lot consisted of four to six specimens, and the mean value of each lot is represented by a single point on Fig. 4, 5, and 6. Large variations in values are indicated by error bars showing greatest and least values.

EXPERIMENTAL RESULTS

General

The void density in one of the samples shown in Fig. 2 is estimated to be of the order of 10^{13}–10^{14}

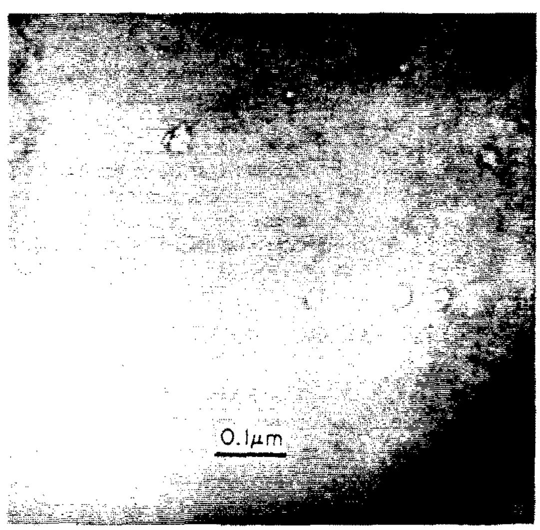

Fig. 2. Voids in heat treated aluminum.

voids/cm^3. The diameters of the voids could not be measured accurately, but rough measurements indicate an average size of about 200–250 Å. The voids were inhomogeneously distributed, and the formation of loops negligible. In many cases, neither the loops nor the voids could be resolved with the aid of an electron microscope, yet a noticeable increase in yield strength was found in the same lot of samples. In such cases, the possible presence of very small voids below the limit of resolution was assumed. Figure 3 shows the structure of one such sample. This sample was slightly damaged in preparation, and the resulting motion of dislocation in the field of the vacancy clusters is seen in the picture. On the basis of the shapes of the curved dislocations in lower half of the area, an arrow (*S*) has been drawn to indicate a possible direction of stress. It appears from this figure that the dislocations are not obstructed by dislocation loops but that they are pinned only by very small voids.

Many void strengthened samples tested in liquid nitrogen at a strain rate of 3.33×10^{-4}/s exhibited an unstable deformation characteristic that emitted an easily audible click similar to that reported by Basinski [12] for aluminium samples of comparable purity and size that were deformed near liquid helium temperatures. He observed no load drops prior to an approx. 8% elongation. In this experiment, the serrated load–elongation curve for the void strengthened samples did not have such a uniform pattern with regard either to elongation prior to the appearance of unstable deformation or to the extent of the load drops, which were sometimes as much as 20% of load.

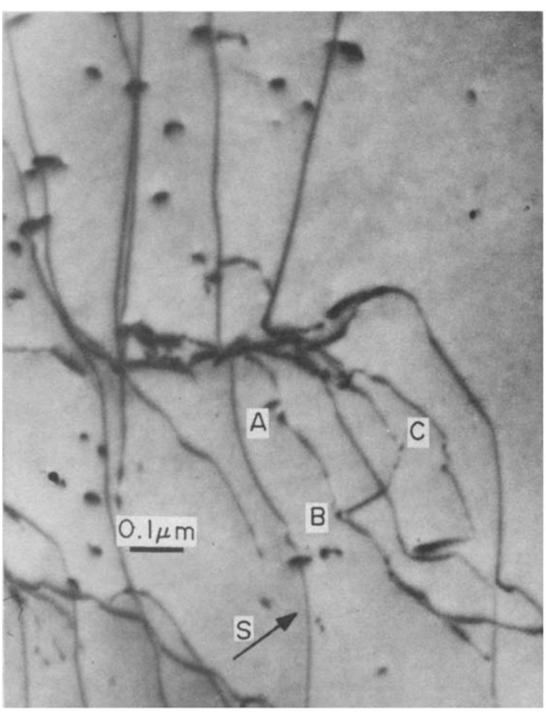

Fig. 3. Dislocation motion in the field of vacancy clusters showing voids pinning dislocations.

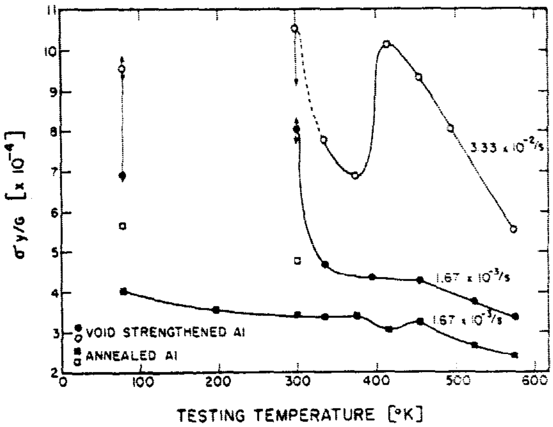

Fig. 4. The temperature dependence of the yield strength.

This can be reasonably expected on the basis of an explanation given by Basinski, i.e. the reduced specific heat for aluminum at 77 K produces a significant temperature rise during deformation that causes a localized reduction in the stress for plastic flow. This instability of plastic flow at 77 K was not observed at lower or higher strain rates nor for the well annealed samples at any strain rate used at 77 K.

Strength and deformation characteristics

Frequently, an inhomogeneous deformation occurs in both large and small areas of a sample under tensile loading. Consequently, the cross head speed is not a true measure of the strain rate; however, for simplicity, the cross head speed is assumed to represent the mean strain rate.

Figure 4 shows the temperature dependence of the yield strength. The strength is rationalized by using the *G* values given by Sutton [13] for the elastic constant, C_{44}. The void strengthened samples show a pronounced temperature dependence of yield strength that changes in different temperature intervals. In general, the temperature dependence of yield strength is nonlinear below 413 ± 20 K and shows a linear variation above about 430 K. The well annealed samples have a much lesser temperature dependence of yield strength.

Other observations. At room temperature, an increase in yield strength resulting from void strengthening is about two and one-half times the yield strength of well annealed samples. This comparison changes considerably in a narrow temperature range just above room temperature. At the high strain rate, 3.33×10^{-2}/s, the variation of yield strength is very uncertain because of the broad spectrum of values at and near room temperature. Because a small variation in temperature elicits a very large variation of strength, there is a greater uncertainty about the strength of a sample in this temperature range. This is shown by the dashed part of the curve.

In the temperature range of 413 ± 20 K, the increase and decrease of yield strength is very marked at the high strain rate, 3.33×10^{-2}/s. At this strain

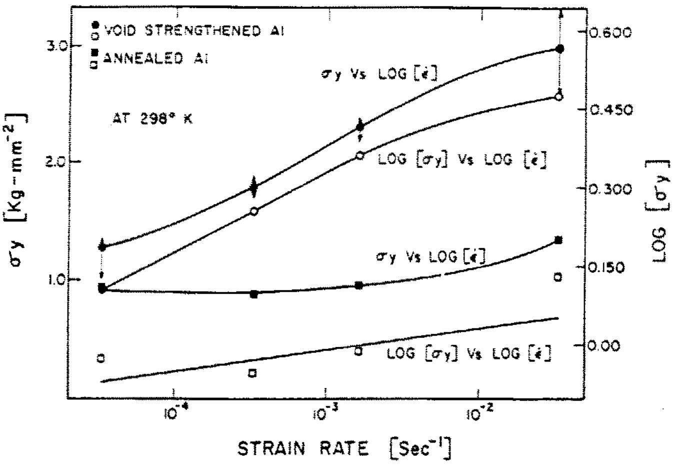

Fig. 5. The strain rate dependence of the yield strength at 298 K.

rate, the yield strength increases sharply to a maximum of about 413 K and then, although decreasing rapidly, remains quite high up to about 500 K. With some dislocation climb being prevented as a result of attractive interactions between voids and dislocations and a thermally activated complementary climb helping to capture a larger number of dislocations by the voids at this strain rate and temperature, the effectiveness of void strengthening seems much improved. With increasing temperature above this range, the thermal activation is predominant and assists the dislocation to escape from the voids.

At the lower strain rate, 1.67×10^{-3}/s, the yield strength is almost constant in this temperature range. At 393 K, two of the six specimens tested at this strain rate showed repeated yield points on the load–elongation curve (the upper one corresponding to the values in Fig. 4). At 433 K, one of the six specimens tested showed a very sharp increase in the load after a large yield point elongation. This and the rise and fall of the yield point at the higher strain rate are considered as indicative of a possible change in the work hardening mechanism; as a result, the authors refer to the temperature 413 ± 20 K as a transition zone. The well annealed samples tested at the strain rate of 1.67×10^{-3}/s showed a slight drop in yield strength in this temperature range. It is noted that Lytton *et al.* [14] have seen a transition in the activation energy for creep in pure aluminum single crystals in the same temperature range.

Strain rate dependence of the yield strength

Figures 5 and 6 show the strain rate dependence of the yield strength of the void strengthened samples at room temperature and at liquid nitrogen temperature, respectively. Similar data that were obtained for the well annealed samples are also shown in the figures. From the empirical relationship, $\sigma = C\dot{\epsilon}^m$, at a constant strain and temperature, the strain rate sensitivity, m, given by the slopes in Figs. 5 and 6, is approx. 0.042 at room temperature and 0.033 at 77 K for well annealed samples. For all of these samples, the sensitivity seems to be much decreased at lower strain rates. In comparison, the void strengthened samples show a very high strain rate sensitivity of about 0.145 at room temperature and about 0.094 at 77 K. The strain rate sensitivity at room temperature (Fig. 5) decreases at the higher strain rate of about 3.33×10^{-2}/s; whereas, at 77 K (Fig. 6) it decreases at a lower strain rate, 3.33×10^{-5}/s.

In Fig. 7, the above data are plotted as increments in the strain rate dependent yield strengths of void

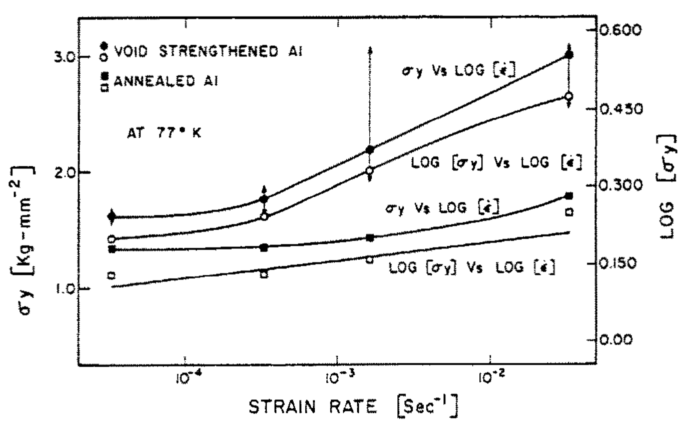

Fig. 6. The strain rate dependence of the yield strength at 77 K.

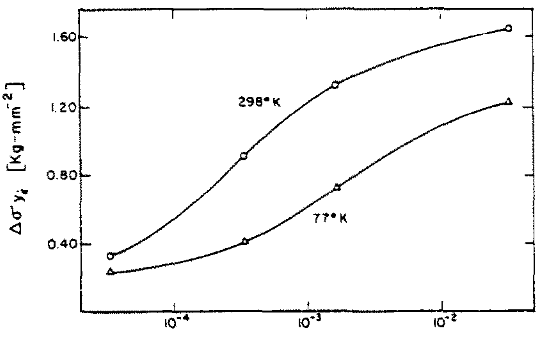

Fig. 7. The increment in strain rate yield strength of void strengthened specimens above the annealed specimens.

strengthened samples above those of the annealed samples. The two curves corresponding to room temperature and 77 K have a strongly inflected S-shape, and the increments in the strain rate dependent yield strength tends to flatten at the lower and the higher strain rates. Hart [15] has explained the strongly inflected S shape of the strain rate sensitivity curve for polycrystalline or multiphase specimens on the basis of the model that the steady state deformation of a polycrystalline material is a flow of a non-Newtonian body (the matrix) containing a dispersion of plate-like flaws (grain boundaries), which permit constrained shear sliding at their surfaces by Newtonian friction. Hence, it is felt that the increased strain rate sensitivity of void strengthened samples may be due to the distribution of additional flaws, i.e. voids in the non-Newtonian matrix.

DISCUSSION

Coulomb, in his theory [4] of void-strengthening, defines the equilibrium shape of a dislocation segment blocked by the voids and calculates the energy of escape for bowing out of the dislocation segment from the voids. According to his criterion, the dislocation escapes from the pinning voids once the dislocation is forced between the voids to bow to the maximum energy position. This maximum energy (escape energy) position is found in terms of the equilibrium angle, θ, made by the dislocation segment at the pinning voids, and the corresponding strain, ϵ. The Coulomb treatment requires the dislocation to remain at the void until it acquires the maximum energy position. Also, it has a large built-in approximation for the values of escape energy and strain that are passed on to the values of stress.

Ashby's treatment [16, 17] of Orowan stress for bypassing the dislocation between the chain of particles envisages forcing the dislocation into a critical shape so that further bowing and snapping of the dislocation from the pinning particles occur as a result of the dislocation's own reaction between the adjacent segments. This approach brings the Orowan stress closer to Coulomb's ideas of dislocation escape from the chain of voids. Therefore, a more precise value of the required stress for dislocation bypassing between voids may be taken from Ashby's dipole criterion on Orowan stress [17],

$$\tau = A\,\frac{Gb}{2\pi}\,\frac{1}{L}\cos\theta\,\ln\frac{x[1 + (L/x - 1)\sin\theta]}{r_0}, \quad (1)$$

in which A is equal to $(1 + \{\nu/(1 - \nu)\}\sin^2\alpha) = 1/(1 - \nu)$ for edge dislocations and to unity for screw dislocations; α is equal to the angle between the burgers vector and the dislocation when no stress is applied, θ to the angle between the arm of the dislocation under stress and the slip direction, x to the diameter of the voids, L to the average void spacing along the dislocation line blocked by the voids, r_0 to the inner cut off radius, and ν, G, and b are poisson's ratio, shear modulus, and burgers vector, respectively.

The shear stress, τ, has a maximum value for θ between 15° and 30°; so for most values of L and x, an arbitrarily chosen value of $\theta = 20$ degrees gives the required shear stress to move the dislocation past the voids as

$$\tau = A\,\frac{Gb}{2\pi}\,\frac{0.94}{L}\ln\left(\frac{0.34\,L}{r_0}\right). \quad (2)$$

For N_v voids/cm^3 of diameter x, Kocks [18] gives the average obstacle spacing,

$$L = \frac{1}{\sqrt{xN_v}} - \frac{\pi x}{4} \quad (3)$$

By using these relations, the expected increase in the yield strength resulting from the presence of an assumed homogeneous distribution of uniformly sized spherical voids can be shown in Fig. 8. According

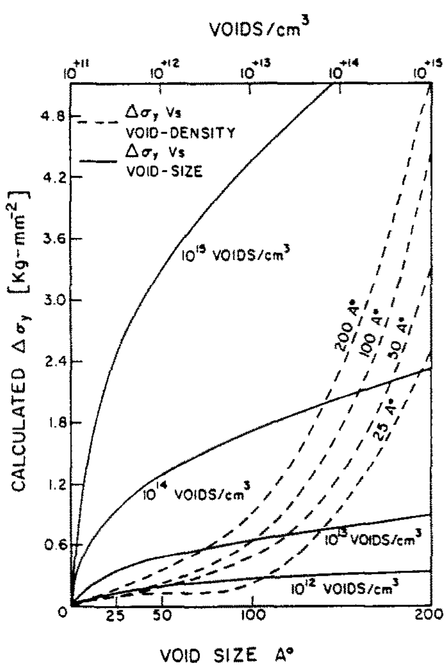

Fig. 8. The expected increase in yield strength as a function of void size and void density.

to the results shown in this figure, a minimum void density of the order of 10^{12} voids/cm^3 is needed to obtain a measurable increase in the yield strength of aluminum. By increasing the void size alone, the increase in yield strength could be restricted by the fact that only voids of a certain maximum average size and density above the necessary minimum value can be accommodated in the volume of metal without forming a larger defect (such as a microcrack), which would affect the strength adversely. Also, the average void size is limited by some competing mechanisms for increasing void density with the available vacancy supersaturation. These two separate limiting factors are not included in the simple model of Orowan stress. Yet, the familiar stress relation seems sufficiently accurate for predicting the extent of void strengthening. By allowing for an inhomogeneous distribution of voids in the experimental specimens, the observed void density was determined to be in the range of 10^{13}–10^{14} voids/cm^3 and the average void size to be about 100 Å in diameter. From this, the predicted increase in yield strength that results from void strengthening as determined from Fig. 8 was determined to be about 1.6 kg/mm^2; whereas the measured vaue of the increase in samples at room temperature was about 1.32–1.65 kg/mm^2 above the yield strength of the annealed samples at corresponding strain rates. The results are in a fairly good agreement with the expected values.

It is to be noted that the dislocation segment may snap away from the voids through its own reaction after bowing further to its critical shape. Therefore, the voids need be just large enough to keep the dislocation segment pinned, until it reaches a critical shape corresponding to the maximum applied stress to bow out the dislocation between the voids. An exact value of the attractive interaction energy between the void and the dislocation is thus unimportant; consequently, the extent of the void-strengthening can be considered independent of the void size distribution.

The smallest (limiting) size of a void that can exert some attractive force on a dislocation that is in contact with it is found by setting the energy of interaction at zero in the solution for the interaction energy established by Week *et al.* [19] for a void of diameter, x, that is symmetrically located on a screw dislocation. From their calculation, the interaction energy is

$$E^{\text{INT}} = [-Gb^2x/4\pi][(\pi^2/12) + \ln(x/2r_0)]. \quad (4)$$

Therefore, for $x \neq 0$, $(\pi^2/12) + \ln(x/2r_0) = 0$ is a limiting case, or $x = 2r_0\,e^{-\pi^2/12} = 0.878r_0$. This indicates that voids as small as the size of the dislocation core may act as obstacles to the dislocation motion. This also suggests that the mechanism and effectiveness of void strengthening are much less dependent on the void size and are primarily governed by void spacing or the prevailing void density, which in the case of aluminum should be above 10^{12} voids/cm^3 to obtain a measurable increase in yield strength.

CONCLUSIONS

1. Similarity between Coulomb's approach to void strengthening and that of Orowan stress with dipole reaction is observed, whereby the extent and effectiveness of void strengthening are primarily dependent on the void-density and much less on void-size. The strengthening obtained at room temperature can be reasonably estimated by the modified Orowan stress model.

2. The observed yield strength in void strengthened aluminum samples shows changing patterns in different temperature ranges. The samples retain more strength than annealed samples over a wide temperature range.

3. Void strengthened aluminum shows a higher strain rate sensitivity of yield strength both at room temperature and at 77 K.

4. Void strengthened aluminum shows increased susceptibility to the thermal instability of plastic flow at low temperatures. This interfaces considerably in obtaining the correct picture of the variation of yield strength with testing temperatures in the low temperature range.

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