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A study of the void-strengthening of aluminum and its nature

Purushottam G. Manusmare

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A STUDY OF THE VOID-STRENGTHENING OF ALUMINUM AND ITS NATURE

BY

PURUSHOTTAM G. MANUSMARE, 1940-

A THESIS

Presented to the Faculty of the Graduate School of the UNIVERSITY OF MISSOURI-ROLLA

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I. ABSTRACT

Strengthening of aluminum by formation of the voids and the nature of resultant strengthening, viz., the temperature and the strain rate dependence of its yield strength were studied. Aluminum wires with 99.999% purity and of 3.1 mm diameter were quenched and heat treated to form voids of the approximate density $10^{13}$-$10^{14}$ voids/cm$^3$ and were tested for their yield strength. Voids in selected samples were observed by transmission electron microscopy using the Hitachi HU-11A microscope. The yield strength of the void strengthened samples was measured at various temperatures from 77°K to 593°K using two strain rates, $3.33 \times 10^{-2}$/sec and $1.67 \times 10^{-3}$/sec and the tests at room temperature and at 77°K were carried out using various strain rates.

The results are examined with the help of a model presented here for the mechanism of void strengthening. The model follows directly from the Orowan stress with Ashby's improved criterion together viewed from the Coulomb's approach. Limitations of both approaches are also discussed.

From the mode, it is shown that the mechanism of void strengthening and the effectiveness of voids as obstacles to the dislocation motion and therefore the extent of void strengthening is primarily dependent on the void density and is much less dependent on the void size.
The results obtained are found to be consistent with the model at room temperature. The void strengthened aluminum samples as compared to annealed samples show following characteristics.

1. They are more susceptible to the instability of plastic flow at low temperatures.

2. Their temperature dependence of yield strength varies in different temperature ranges; and

3. They have higher strain rate sensitivity of yield strength.

These observations are explained in terms of various reactions occurring simultaneously.

In a separate part, an hypothesis is presented to suggest that a thermodynamic cycle may be operating in a rotating component under load and therefore its fatigue life is some function of the strain rate sensitivity of both the material and its bulk thermal conductivity.
ACKNOWLEDGEMENT

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I. INTRODUCTION

More than forty years have passed since Frenkel (1926) first proposed the generation of an equilibrium number of vacant lattice sites and interstitial atoms by thermal fluctuations in solids. Soon detailed models of the defects in special cases were presented by various workers but the nature of these thermally induced defects has remained a matter of speculation and controversy for a long time because of a noticeable lack of experiments that allowed differentiation between the various kinds of defects. It is apparent that the behavior of quenched-in vacancies and even the production of vacancies are still not completely understood. A somewhat clearer picture of the large clusters of vacancies is now emerging due mainly to the fact that it has become possible to observe some of these clusters by electron microscopy. However, the size of vacancy clusters observable in the electron microscope is limited by its resolving power, so the uncertainty in the correlation of the data obtained by other techniques persists. In recent years the field ion microscope, though its applications are limited to the high temperature metals, has been developed for configurational studies of the vacancy clusters.

While relating the effect of vacancy clusters on the properties of solids, their description in the literature prior to 1960's is somewhat ambiguous at times. Formation
of di-, tri- or tetra vacancies, condensation of vacancies into the dislocation loops and also into stacking fault tetrahedra, of which only the last two were observed by electron microscopy for the first time in 1958 and 1959. Three dimensional vacancy clusters with some form of assumed surface have been regarded to be cavities or "voids". Since then, the nature of these defects has been sought in order to understand radiation damage, for the study of self diffusion and for the measurement of surface energy. For studying these defects in metals, the quenching method has some advantages as follows:

a. Unlike irradiation procedures, the major point defects created at high temperature are vacancies which are trapped without much loss in number during quenching. Formation of different kinds of vacancy clusters by different post quench treatments can then be immediately followed or probed by suitable means.

b. Unlike cold working, a large number of dislocations are not created.

The existence of the vacancy clusters mentioned above had been postulated by the early 1950's (sessile dislocations by Frank in 1949, dislocation loops by Seitz in 1950 and voids by Pratt in 1954). These vacancy clusters were characterized in such a way as to explain their effect on the properties, particularly the mechanical properties,
of crystals which depend on the existence and motion of dislocations. The complications of dislocation reactions in solids have become progressively clarified in recent years, a part of which can be attributed to attempts to explain the radiation hardening and quench hardening in pure metals on the basis of dislocation reactions with these vacancy clusters. As suggested by Maddin and Kimura\textsuperscript{5}, the tension test is considered to be not only a suitable test for studying the interactions of dislocations with various forms of defects but also to be a simple indirect technique to investigate the fundamental properties of quenched-in defects (i.e. activation energy of formation, activation energy of migration, etc.). However, their effect on mechanical properties other than the yield stress is much less well known partly because of a paucity of data, partly because of a lack of full understanding of work hardening mechanisms in pure and annealed metals, and partly due to the absence of clear identification of the defects responsible for the observed hardening or the extent of their share. It is therefore hoped that the study of strengthening of aluminum by voids being reported here will be of some value in these regards.

The difficulty in correlating the observed strengthening effect with a single type of defect is the result of the difficulty in producing only that type of vacancy clusters. This results in some uncertainty in finding the
cause of the total strengthening observed. Under this situation, the choice of pure aluminum to study void strengthening in metals seems to be the most appropriate one because:

a. With proper thermal treatment it has become possible to create a considerably greater number of voids than loops, these two being the predominant vacancy clusters produced by quenching aluminum.

b. Reasonable assumptions can be made on the basis of available information regarding the stability, the formation and the annealing behavior of these defects.

c. The available information can be extended to the void-strengthening reactions, and

d. Previous data on the quench-hardening of aluminum can be used for comparison.

It may not be out of place to say here that the voids formed in radiation damage differ from the thermal voids with regard to the possible association of gases within them and the occurrence of reaction product behavior which may be a complicating factor. Nevertheless, the literature relating to the radiation voids is an important source of information.
II. LITERATURE REVIEW

A. General Background

Progress in the study of void strengthening in crystals in general and metals in particular can be followed in its development along the two directions:

a. Advances in the knowledge of the structure of quenched-in defects in metals, and

b. Progress in updating their correlation with the observed mechanical behavior.

Pratt\textsuperscript{4} put forward the idea that void strengthening is a possible cause of the observed strengthening of quenched rock-salt crystals, but the extent of such a strengthening was first calculated by P. Coulomb\textsuperscript{6}. In 1957 Coulomb\textsuperscript{7} assumed the possibility of formation of cavities during irradiation and, on the basis of image forces, he calculated the attraction exercised by these cavities on dislocation lines. Later, in 1959, Coulomb\textsuperscript{6} went on to calculate the increase in the strength of the crystal along with the temperature dependence of the yield strength in the case of void strengthening. By electron microscopy and by using the electron beam to move the dislocations nearer to the voids A.J. Forty\textsuperscript{8} observed the attractive interactions between voids and the dislocations in a lead
iodide crystal. No such direct observations of interactions between the moving dislocations and the voids in metals have been made, although only the attractive interactions are expected and used by various workers to correlate the radiation hardening with the observed or postulated voids.

Early observations of vacancy clusters as quenched-in defects is due to Hirsch et al.⁹ who found the dislocation loops resulting from the aggregation of vacancies in quenched aluminum foil. They discussed this in relation to the problem of annealing of vacancies, of quench-hardening and of the origin of the dislocation networks. Smallman and Westmacott¹⁰ in their observation of neutron irradiated aluminum found jogged and spiral dislocation loops. The presence of loops in quenched aluminum was then considered to be responsible for the earlier experimental observations of Maddin and Cottrell¹¹. They had found that the stress-strain curve of aluminum crystals quenched from near their m.p. differed significantly as regards to the increase in yield stress and the region of low work hardening after yield point when compared with the behavior of well annealed samples. This was explained by them as being caused by the quenched-in vacancies. These authors had noted an age hardening character of quenched aluminum suggesting an agglomeration or the movement of vacancies to certain positions and not just the random distribution of vacancies to be
responsible for the phenomena.

However, Wilsdorf\textsuperscript{12} found no such observable defects, i.e. loops, in neutron irradiated nickel to explain the observed increase in its yield strength. Kuhlmann-Wilsdorf et al.\textsuperscript{13} acknowledged the possible existence of voids, viz. three dimensional vacancy clusters essentially empty inside, as another source of hardening. Also Kuhlmann-Wilsdorf and Wilsdorf\textsuperscript{14} postulated voids to be an intermediate step in the formation of the observed loop pairs and loops of unexpected shapes and orientation found in quenched aluminum. Later, based on detailed studies on neutron irradiated copper, it has been suggested by Makin et al.\textsuperscript{15} that the major contribution to the hardening is not by the observable dislocation loops but by the sub-microscopic defect clusters below resolution of the electron microscope. Direct observation of the presence of voids in quenched metals was made later in quenched platinum foil by Ruedl et al.\textsuperscript{16}. In their observation some voids were seen interacting with (presumably pinning) the midsections of dislocations. These voids disappeared only after annealing at about 800°C.

Similar small (20-100 Å) vacancy clusters, i.e. voids, were reported by Kiritani and Yoshida\textsuperscript{17}. The extensive study of the formation of these vacancy clusters (voids, loops and stacking fault tetrahedra) in quenched aluminum was carried out by the Japanese school\textsuperscript{17-28}. More of this
work will follow in part B of this section.

In the meantime, the studies on quench-hardening in aluminum were repeated with a slight change. Maddin and Kimura\textsuperscript{5,29} and Shiotani et al.\textsuperscript{30} maintained that the observable loops are responsible for quench-hardening in aluminum explaining that the jogs may also be contributing to it. Initially Westmacott\textsuperscript{31} concluded that voids are responsible for the quench-hardening where small temperature dependence of the yield stress was observed. However, in 1967 Westmacott\textsuperscript{32} came to a different conclusion that very small loops, viz. vacancy discs, of 6-7 vacancies were responsible for the quench-hardening when the temperature dependence of the yield stress, given by Fleicher's theory\textsuperscript{33,34} was found to be obeyed in his experiments. It is indeed difficult to resolve their differences at this stage, although further comments are made in part C-3 of this section and in the discussion.

B. Voids in Quenched Aluminum

1. Formation of voids

In studying the strengthening of aluminum by voids, the formation of voids and their size and distribution is of prime importance. The experiments on the nucleation and growth of the voids in aluminum have been carried out almost exclusively by the Japanese school under Prof. Yoshida\textsuperscript{17,20,21,23 to 28}. In their experiments, voids and
loops have been found to form together where their proportion of voids to loops can be varied within certain limits by careful manipulation of the heat treatment parameters. Following Kiritani\textsuperscript{20}, a high pre-quenching temperature combined with slow quenching rate and higher aging temperature was found to promote the void formation in relation to the loop formation. The schematic diagram of the heat treatment of Kiritani\textsuperscript{27} and the density of voids and dislocation loops obtained thereby is reproduced in Figs. la and lb. The time for the initial aging in this treatment was short and very critical to obtain higher density of voids with respect to loops. Therefore the procedure required a two layer bath in which the specimen remains for the critical time in the top layer maintained at lower temperature before entering into the bottom layer maintained at higher temperature for the final aging treatment of more than 10 hours.

Shimomura and Yoshida\textsuperscript{28} showed that the voids are preferably formed by quenching in atmospheres of hydrogen and wet air and no observable voids were formed under other atmospheres of CO, dry air and vacuum. This was interpreted by them as the heterogeneous formation of all voids by the dissolved hydrogen acting as nuclei. It is interesting to note that the maximum density of voids thus obtained under a hydrogen atmosphere is the same as that obtained by Kiritani without the use of any special atmospheres.
Fig. 1a: Schematic Diagram of the Heat Treatment for Void Formation [Kiritani27]

Fig. 1b: Density of Voids and Dislocation Loops Obtained by the Heat Treatment [Kiritani27]
Westmacott\textsuperscript{35} mentioned that workers in other laboratories have been less successful in producing a high density of voids in this manner and attempts to standardize the quenching conditions by passing wet air through the furnace during the pre-quench heating were only partially successful. Other reported experimental set-ups for void-formation were in connection with annealing studies on the thermal voids where high density was not a primary concern. However, the evolution of a simpler experimental set-up as given in the experimental procedure, C-2, for creating the voids in the present study is based on the above described studies.

2. Nature and observation of voids by electron microscopy

A number of factors that determine the image contrast for the voids in aluminum have been fairly well discussed by Kiritani\textsuperscript{20}, Kiritani et al.\textsuperscript{21} and for small defects in general by Bell et al.\textsuperscript{36}. In principle, the contrast of the void image is caused by the reduction of effective thickness of the foil due to the presence of void. The beam intensity of the region containing the voids thus deviates towards the intensity of the thinner part and then, depending on the diffraction contrast, the images of voids are seen as black or white spots against the contrasting background. The clarity of image can vary considerably according to the defect size and its position
relative to the foil thickness as shown on the beam intensity vs. the foil thickness curve, figure 2.

The loops are seen by strain contrast, so in a little thicker region where the dislocation loops can still be seen, the observation of voids may be quite difficult, because the difference in the beam intensity caused by the effective thickness change is reduced for these thicker areas. This will affect the density measurement of voids in such areas. Also voids of small sizes situated at the flat part of the curve in the figure 2 (location and size shown by A and B) will not cause enough intensity contrast and so sometimes only some of the strain field will be observed.

The intensity distribution of the image of voids is very much dependent on its size, shape and its inclination with respect to the incident beam as explained by Kiritani et al.21. The void image in many cases will be smaller than the actual size of void and the shape of the image does not indicate the real projection of the void unless fine contrast conditions are obtained.

The image contrast between voids and the loops can be differentiated by the following rough guidelines besides those given above.

a. The voids being three dimensional clusters, their images do not disappear by tilting the specimen to change the diffraction conditions.
Fig. 2: Schematic Diagram of Beam Intensity vs Foil Thickness
b. Image contrast of loops has a directional structure while the void images can have an irregular contrast with the background in case of an uneven specimen surface.

3. Stability of voids

Relative stability of the various forms of vacancy clusters have been treated theoretically by many workers using elastic theory as well as atomistic models. The energy calculations have been recently summarized for nickel by Laidler\textsuperscript{37} using earlier data from Johnson\textsuperscript{38} and have shown that the voids and tetrahedra are more stable than the dislocation loops and other shapes of vacancy clusters. Similar energy calculations for aluminum by Sigler and Kuhlman-Wilsdorf\textsuperscript{39}, and the experimental observation of Westmacott\textsuperscript{35} on the void-formation caused by short time post quench aging at 150°C suggest the enhanced stability of voids. The stability of the void nucleus is apparent from the experimental observations of Kiritani et al.\textsuperscript{27} and from the atomistic calculations of Cotterill and Doyama\textsuperscript{40}.

On the basis of these calculations, it seems that the formation of void-nuclei (the nuclei being very small) is not influenced by the stacking fault energy or the specific surface energy of the metal. By appropriate combinations of quench parameters, the formation of void-
nuclei could be expected in any monomorphic pure metal. However, the voids are not observed universally indicating that the stability of voids is greatly influenced by the growth parameters (which are much less well understood) including impurity effects. In case of aluminum wherein the formation of voids is already known to occur, the present interest is then confined to the stability of voids after they have been formed.

4. Bulk annealing of voids

The reported studies of Volin and Balluffi\textsuperscript{41} and that of Westmacott and Smallman\textsuperscript{42} on the annealing of thermal voids in the electrothinned samples cannot be related to the bulk annealing of voids. Shimomura\textsuperscript{23} performed bulk annealing of the secondary defects in 60\textmu thick quenched aluminum foils. He reported the possibility of the growth of voids up to 160°C and void shrinkage to begin at about 170°C. The voids disappeared by annealing at 220°C for 15 minutes. Also the void shrinkage was observed to be retarded when they are present in the region of Frank sessile loops with the loops annealing out considerably faster and at comparatively lower temperatures. The bulk annealing behavior of voids is expected to be strongly influenced by the specimen thickness and proximity of grain boundaries and loops.
Westmacott and Smallman\textsuperscript{43} observed that the bulk annealing time for radiation induced voids in 1/8 inch diameter disc specimens (prior to thinning) at 195°C was 25 times greater i.e. 120 hours, than that for the voids in the electropolished specimens. Shrinkage rate of the voids was also found to be dependent on the nature of the specimen surface and the electron beam irradiation received by the same.

It is known that some gas molecules will be located within the voids, the amount of gas present being considerably more in radiation voids. Westmacott and Smallman\textsuperscript{43} have suggested that the stability of voids originates in the difficulty in outward diffusion of molecular hydrogen (requiring dissociation before the diffusion) into the lattice where the re-solution of hydrogen atoms at the cavity-lattice interface is the rate controlling step in the process of void shrinkage. The stability of voids in the bulk annealing of such aluminum samples can best be tested by creep tests.

C. Strengthening Effect of Voids in the Crystalline Solids

An examination of the Coulomb model of void strengthening suggests that a better approach to the problem of void strengthening can be made on the basis of Ashby's\textsuperscript{44} refinement of what has been known as the Orowan stress in dispersion strengthening. Neither Ashby nor Orowan considered
void strengthening. The recent and more extensive analysis of the elastic interactions between a straight dislocation and a spherical bubble or a particle has been given by Weeks et al. but it cannot be used here directly. However, the extension of their results in connection with the consequences of this study is also given in the discussion.

1. Voids as obstacles to the dislocation motion.

Coulomb's treatment of void-strengthening is fairly simple except for the geometric complexities involved. He defines the equilibrium shape of a dislocation segment blocked by the voids and calculates the energy of escape for the bowing out of the dislocation segment from the voids. The criterion used by him for the escape of the dislocation segment from the pinning voids is that, due to the applied stress or thermal agitation, the dislocation segment bows to a critical shape (escape position) which corresponds to the maximum energy position of the dislocation between the two voids. Then two possibilities of escape are considered. In the first case, the escape between the two voids is considered, and in the second case, the escape between three voids involving an intermediate step of the dislocation forming an excited position over the middle void such that it still maintains the equilibrium configuration between consecutive voids. Then the escape from these two voids is similar to the escape in the two voids case. This second possibility appears to be more common.
The shape of the dislocation after attaining the escape energy which is considered by Coulomb to be the maximum energy, $\Delta E$, of the dislocation segment has been used to find the corresponding strain, $\varepsilon$, produced and these two quantities are then related in terms of the equilibrium angle, $\theta$, made by the dislocation segment at the pinning voids. For the escape between two voids, he obtains

$$\frac{\Delta E}{Gb^3} = \frac{1}{4\varepsilon} [\pi - 2\theta - \sin 2\theta]$$

and for the more general case of the escape between three voids, a longer relationship takes the approximate form

$$\frac{\Delta E}{Gb^3} = \frac{r}{b} \alpha [1 - 2\beta \frac{b}{l} \varepsilon]$$

where, $r =$ radius of voids

$L =$ the spacing between the voids

$b =$ Burgers vector

$\alpha$ and $\beta$ are constants changing their values very slowly for different 1/4 ratios.

The stress required to push the dislocations through the array of voids is then found by using the relationship of the shear modulus, $G$, with the strain, $\varepsilon$.

The model proposed by Coulomb was not expected to hold good for voids very far apart ($1/r > 25$) for which the estimated $\Delta E$ is very small and for voids quite close ($1/r << 3.75$) where a larger error in the approximation of the initial equilibrium shape occurs.
The evaluation of $\Delta E$ is not easy, so Coulomb obtains a final relationship for strain, $\varepsilon$, by equating this escape energy, $\Delta E$, with the activation energy for slip from strain rate relationship. This yields an expression showing yield strength variation as a function of temperature. A closer look is given to this aspect in the discussion.

The Coulomb treatment may be regarded to be the best approach to the problem, though it has two major flaws:

a. 1. If the pinning of a dislocation by the voids is not strong enough, the dislocation segment should be expected to liberate itself from the voids before it is stressed to the maximum energy configuration, or

2. If the attractive energy between the voids and the dislocation is much stronger than that corresponding to the maximum energy configuration of the dislocation segment, then two possibilities arise,

i. an isolated dislocation segment pinned by the two voids will act as a Frank-Read source, and

ii. if the dislocation is blocked by a chain of voids, the interaction between the adjoining bowed segments will lower the escape energy to a value below that of the assumed maximum $\Delta E$ without such interaction (as in the case of Coulomb's criteria).
b. It has a large built-in approximation for the value of strain, $\varepsilon$, which is passed on to the value of stress.

Without deviating from the Coulomb approach, these flaws could be corrected by using the improved criterion of Ashby\textsuperscript{44} on the Orowan stress for bowing out of the dislocation segment between dispersed particles. The two approaches could then be directly merged to give the increase in shear stress as

$$
\tau = A \frac{Gb}{2\pi} \frac{0.94}{L} \ln \left( \frac{0.34L}{r_o} \right)
$$

(1)

Where

$$
A = (1 + \{\mu/l - \mu\sin^2 \alpha\})
$$

= $1/l - \mu$ for edge dislocations.

= 1 for screw dislocations

$\alpha$ = angle between the burgers vector and the dislocation when no stress is applied.

$\mu$ = Poisson's ratio

$G$ = shear modulus

$b$ = Burgers vector, and

$L$ = average void spacing along the dislocation line blocked by the voids.

$r_o$ = inner cut off radius.

The merits of these calculations as offering a better model of the void-strengthening mechanism are given in the discussion. These approximations
are very close to the computerized calculations of A.J.E. Foreman\textsuperscript{46}.

A large uncertainty in the value of the applied stress can be expected from:

a. The variation of the constant, $A$, which changes with the nature of the dislocation, and

b. The variation of void-spacing, $L$, for which no proper averaging is possible.

2. Remarks on the void-spacing

The determination of the void-spacing, $L$, is not a trivial problem because it can be an important quantity in any attempt for the quantitative verification of the hardening theory by experimental results of a study of strengthening by randomly dispersed particles of voids. U.F. Kocks\textsuperscript{48,49} discussed this problem as a statistical theory of alloy hardening. The obstacle spacing has been discussed by many authors including Dorn et al.\textsuperscript{50}, T. Suzuki and Ishi\textsuperscript{51}, Westmacott\textsuperscript{52} and others.

Amongst the many statistical averaging methods for calculating the average obstacle spacing, $L$, a simple one is the square root of the mean area per obstacle-point in the slip plane minus the mean obstacle diameter in the slip plane. This was given by Kocks\textsuperscript{48} as
where \( c \) is the volume fraction of spherical voids of uniform radius, \( r \). If \( N_v = \) number of voids per cm\(^3\), i.e. void-density, this yields

\[
L = r \left\{ \sqrt{\frac{2\pi}{3c}} - \frac{\pi}{2} \right\}
\]

3. A brief comment on the loop strengthening

H.G.F. Wilsdorf and D. Kuhlman-Wilsdorf\(^5\)\(^3\) showed that a small amount of deformation produced more dislocation tangles in an aluminum sample containing quenched-in loops than compared to that in annealed samples. This was also observed in the present study. However, the "sweeping up" and "sucking in" of the dislocation loops by a moving dislocation was observed in neutron irradiated copper by Makin\(^5\)\(^4\), Sharp\(^5\)\(^5\) and others and the prismatic glide of the loops to the surface by interaction with moving dislocations was observed by Strudel and Washburn\(^5\)\(^6\) and Foreman and Sharp\(^5\)\(^7\). This indicates that the loop-hardening will be somewhat ineffective at higher stress. The earlier theory of loop-hardening by Fleischer\(^3\)\(^3\),\(^3\)\(^4\) considers about 2/3 of the loops to be effective while Westmacott considers about half of them to be effective towards the hardening. Shiotani et al.\(^3\)\(^0\) and Maddin and Kimura\(^5\),\(^2\)\(^9\) have considered
all of them to be effective. Leaving this aspect, the review of loop-hardening in their discussions is still adequate but for the evidence shown by Westmacott\textsuperscript{32} and Makin\textsuperscript{58} that the observed dislocation loops are not responsible for the hardening in their samples; quenched aluminum and neutron irradiated copper respectively.

Any theoretical treatment of loop-hardening without consideration of the crystallography of the loops and their movement under stress is bound to be the most approximate. In one such case, using a digital computer A.J.E. Foreman\textsuperscript{59} has considered the junction reaction hardening by loops on (110) plane by dislocations moving on (111) plane in F.C.C. crystal.

The formation of easily observable loops in aluminum of comparable density together with the formation of voids (not so easily observable) has given rise to much of the controversy about the vacancy clusters primarily responsible for the quench-hardening in aluminum. Assuming an equal opportunity for the growth of all void nuclei formed during this quenching technique (under B-1 of this section) a specific attempt which produces fairly large voids and in which the production of very small voids may be considered negligible, leads to a structure nearly free of dislocation loops, and in which the vacancy concentration is almost exclusively contained in the voids. The strength of such aluminum samples is investigated in this study.
III. EXPERIMENTAL PROCEDURE

A. Material Preparation

The aluminum rods of 99.999% purity with known impurities of Cu 2 PPM, Iron 5 PPM, Mg 0.1 PPM and Si 1 PPM were supplied by the United Mineral and Chemical Corporation, New York. It was rolled to 4.8 mm rods and finally swaged to get the 3.1 mm diameter wires. The specimens each about 6 cm long were cut and annealed at 500°C for one hour before storage.

Maximum care was taken to avoid contamination of the material during its preparation. However neutron activation analysis of the final sample showed a sharp rise in iron content to about 30 PPM.

B. Thermal Treatment for Void Formation

The formation of voids has been found to be very sensitive to a number of quenching variables, and for good reproducibility of results, a sophisticated quenching technique similar to that first used by Kiritani\textsuperscript{20} is required. Kiritani\textsuperscript{20} and Kiritani et al.\textsuperscript{27} have shown that the prequench heating temperature, the quenching rate, and the initial as well as the final aging temperatures are the most important quenching parameters requiring proper control to achieve a suitable void-formation. They achieved this by quenching the samples in a two layer bath.
as briefly discussed in the earlier section IIB-1. Except for a few mechanical alterations made by others the technique has remained basically unaltered.

A significant improvement made by Westmacott in the free fall technique and a brief mention by him about using a 50% Ucon solution in water as a quenchant to create a few big isolated voids have helped to make the experimental set up much simpler for the present studies. He heated each specimen in a cylindrical stainless steel core which maintained the specimen at high temperature during its fall through the furnace until the specimen was released at a level just above the quenching bath. Because the specimens were longer in the present experiments, the vertical fall technique was modified to suit a horizontal fall of the specimens into the quenchant beneath. This also made it possible to heat-treat more specimens (4 to 6) at a time.

In this modification as shown in the schematic diagram, Fig. 3, the specimens were heated in the longitudinal groove of the cylindrical stainless steel core having a long handle. The stainless steel core is 28 cm long with 2.54 cm diameter and has a longitudinal groove 0.95 cm wide, 20 cm long. After heating the specimens in the groove (prequench heating) for about 2 hours the hot core containing the samples is drawn out of the tubular furnace as shown in the photograph, Fig. 4, and the groove is turned
Fig. 3: Experimental Furnace Set Up (Schematic)

Fig. 4: Experimental Furnace Set Up Photo
upside down by rotating the handle fixed to the core. The hot samples fall into the quenching bath placed beforehand under the hot core. The temperature of the core heated to 640°C drops by less than 5°C during this entire operation giving an effective quenching temperature of about 635°C.

With the above arrangement various quenching baths including the two layer bath technique of Kiritani were tried without obtaining enough void-formation. Most of the previous workers have used the technique for foil samples (~150μ thick) whereas for the present studies it was necessary to have 3.1mm diameter wire samples to be tested in tension subsequently. This size difference of samples required a suitable manipulation of the quenching parameters. The Ucon* solution in water was found most convenient for such a manipulation. Secondly, it was felt that a simplicity of procedure could be achieved by using only a single layer bath, i.e., for the initial aging only. So the samples were quickly taken out from the quenching bath (in a wire mesh tray), washed in suitably hot water (depending on the final aging temperature) and aged outside the bath.

*From the reported information60, it is understood that in the elevated temperature range the Ucon separates from the water to form a heat conductive film on the quenched specimen; the thickness of the film is nearly inversely proportional to the temperature thus giving fairly uniform quenching rate. A change in the concentration of Ucon causes a change in the quenching rate.
In the earlier operations it was found that some of the samples had cracked longitudinally after a quench. To sort out such structurally defective samples a pre-quench treatment was given in which the samples heated to $600^\circ \text{C}$ were quenched into a water bath at room temperature. This was repeated 5 times. The total heating time during this process was maintained at $\approx 6$ hours. Some of the samples showed the cracks after 3 quenches and were rejected for further treatment of void-formation.

In trials, after the prequench heating at $635^\circ \text{C}$ for about 2 hours the specimens were quenched in Ucon solution. They were quickly removed from the solution, washed in hot water and transferred to an oven for the final aging. Various Ucon concentrations from 10% to 60% Ucon in water at room temperature were tried for the initial quenching bath. The final aging temperatures were varied from $60^\circ \text{C}$ to $140^\circ \text{C}$ with the final aging time arbitrarily fixed at 5 hours. The intermittent hot water wash of the same temperature as the final aging temperature up to the boiling point of water was used which also served as a transfer medium from the initial quenching bath to the final aging in an oven. Electron microscopy was used to observe the voids in two samples from every bath and the rest were tested in tension. On the basis of a suitable void-formation and the reproducibility of results the final treatment for void-formation was fixed.
as follows. Quenching temperature 635°C with heating time 2 - 2 1/2 hours, initial aging (quenching) in a 40% - 45% Ucon in water at 25°C, the wash water at 95°C - 100°C, and final aging at 105°C - 110°C for about 4 1/2 hours. The time lapse between the initial and final aging was about 15 seconds.

C. Specimen Preparation: Electron Microscopy

The procedure for observing the voids by electron microscopy and for the tensile testing of the void-strengthened samples (briefly described later) are fairly routine ones and therefore only the necessary details or the novelties are described here.

1. Specimen cutting

The suitable specimens for electron microscopy were produced from the samples by slicing about 0.4mm thick disc shaped specimens and then electropolishing them by a jet polishing technique.

The specimen discs were cut from the sample length by using an abrasive wire saw manufactured by South Bay Technology, El Monte, California. This saw is shown in the photograph, Fig. 5. The sample to be sliced is first cemented to a graphite plate (having an aluminum plate backing) using the Eastman 910 adhesive. The cemented sample is then fixed on the micrometer driven stage,
Fig. 5: Abrasive Wire-Saw
brought under the wire blade and the disc shaped specimens are sliced by the fast moving wire saw fed with an abrasive slurry. Two sizes of saw blades, 0.005" and 0.010" diameter stainless steel wires, supplied by the same company, were used. The abrasive slurry consists of about 70 grams of 600 mesh silicon carbide powder mixed in 100 ml of glycerine and 10-20 ml of water. The slurry is kept well stirred in the attached container from which it is continuously fed at the cut by means of a tube pump supplied with the machine. To separate the cemented sample from the graphite plate it has to be soaked in N-Dimethyl formamide for about 2 hours.

This method of cutting the specimen discs is quite suitable as it combines speed with only a very small amount of extraneous surface damage. The operation requires some practice and attention to a proper speed and tension in the wire blade without having undue weight on the blade. A slow speed of the wire between the 40 to 50 marks on the arbitrary scale with very little weight on the wire was found to be best suited for the purpose.

2. Electropolishing

The electropolishing of the disc shaped specimen of about 0.4 mm thickness and 3.1 mm diameter was carried out by a simple single stage, twin jet process described by Hacking et al. and the similar process used by
Westmacott. They have described a jet polishing process without immersion of the specimen in the electrolyte. However, with the available apparatus, the dual jet polisher (Astromet) manufactured by the Precision Scientific Company, Chicago, having the jet diameter 3.18 mm with jets 38 mm apart, the immersion process was tried and found to be quite suitable.

A teflon sample holder with 2.4 mm hole and a platinum wire electrode was made as described by Hacking et al. In operation, the specimen disc is placed between the holes of the two plates of the holder and the platinum electrode touching the specimen is made the anode. The assembly is lowered into the electrolyte and the electro-polishing continued with the electrolyte jets flowing against the specimen until the sample perforates. The sample is then removed and washed thoroughly with distilled water. The composition and the conditions of the electrolyte are as follows:

- Acetic acid 4 parts
- Phosphoric acid 3 parts
- Nitric acid 2 parts, and
- Water 1 part.

Temperature of the electrolyte is maintained below -5°C with voltage about about 25–30volts. The jet flow is maintained at about 60 to 70 marks on the arbitrary
scale and the electrolyte temperature kept between -5°C to -15°C to avoid any unwanted increase in its viscosity. For inspection, the specimen is taken out of the electrolyte without stopping the current flow. When the first perforation appears, the holder plates are unscrewed and the specimen taken out and washed in distilled water. Some electrolyte is always present in the holder which leaves etch marks on the specimen. Therefore, after washing the specimen with the water it is again dipped for 4-5 minutes in a mixture of 35% phosphoric acid and 65% chromic acid and washed clean in the distilled water.

The fresh electrolyte does not work very well; therefore, either some used electrolyte is mixed with the fresh or a small amount of copper or aluminum is added to the electrolyte before the initial use.

This electropolishing technique for aluminum is fast and very convenient for the disc shaped specimens. The technique does not produce any thermal or deformation damage of the specimens. The thinned area at the center of specimen is supported by a thicker annulus, so the specimen handling is more convenient and the specimen is self supporting in the specimen holder of the electron microscope.
3. Electron microscopy

The specimens were examined for the voids by the HITACHI-HU 11A electron microscope operated at 100 KVA. The nature and observation of voids by electron microscopy has been briefly described in the section IIB-2. The larger voids in the thin regions of the foil could be clearly identified in the micrographs but some of the smaller voids are not visible at magnification up to 30,000X. They become clear in the enlarged print of a properly focused plate (negative).

D. Tensile Testing

The Instron model TTD-L was used to measure the yield strength of the samples as a function of the testing temperature and strain rates at room temperature as well as at liquid nitrogen temperature. The gauge length was maintained at 1" and the yield stress was taken as the 0.1% proof stress from the load-elongation graphs. For the strain rate $3.33 \times 10^{-2}$/sec the change in slope of the chart trace corresponded to the 0.1% off set strain and so the stress at the sharp change in slope was taken as the yield stress.

At low temperatures below room temperature, the specimens were stressed in a tubular attachment to the moving crosshead. In this arrangement the specimen is stressed by moving the tube downwards while cold liquid inside the
tube is maintained at a high enough level to keep the specimen immersed in the liquid. A mixture of dry ice and methanol was used for the tests at 195°K and liquid nitrogen was used for the same at 77°K.

For testing at higher temperatures the samples were tested in a split type tubular furnace.

The tension tests of the samples were made at eight or nine distinct temperatures from 77°K to 593°K. Each test lot consisted of from four to six specimens. The experimental points plotted on Figs. 10, 11, and 12 (pages 43, 47 and 48) each represent the test values from one such lot of specimens. The error bars on these three figures represent the greatest and least numerical values found at each test temperature. Data for a typical run and sample calculations are shown in Appendix D. Results for a total of approximately 250 samples are represented by the experimental values shown on the three above mentioned figures.
IV. EXPERIMENTAL RESULTS

A. The Formation of Voids

Non-uniform grain size of the different specimens posed a problem from the beginning. The grain size of the material after rolling, swaging and then annealing for one hour was quite uniform but the grain size of the long annealed samples and the void-strengthened samples varied greatly even within the same test sample. Counting the number of grains revealed by cutting along the specimen axis, the grain size varied from 0.5 mm to 7 mm.

Figure 6 shows the voids in one of the samples. Assuming the observed area to be about 3000 Å thick with the void-free surface of about 250 Å thickness on each side, the void density in the area is estimated to be of the order of $10^{15}$ voids/cm$^3$. The image contrasts and shapes of the voids are in as good agreement for octahedral voids in different orientations with respect to the incident beam as in the work of Kiritani et al. Some void-images of the type shown by them as being difficult to explain were also observed. These are shown as voids A and B in Fig. 7. The distribution of voids was very inhomogeneous with a negligible formation of loops. In many cases neither the loops nor the voids were seen in the microscope yet a noticeable increase in yield strength
Fig. 6: Voids in the Heat-Treated Aluminum Sample \( 1\text{cm} = 1110\AA \)  

Fig. 7: Voids A and B Show Complicated Images \( 1\text{cm} = 1050\AA \)
was found in the same batch of samples. In such a case, the possible presence of very small voids below the limit of resolution was assumed. Figure 8 shows structure of one of the samples from the specimens treated to form voids by the double layer bath technique. Apparently it has resulted in forming a good number of observable loops and possibly some very tiny voids. This sample was slightly damaged and the resultant motion of dislocations in the field of such vacancy clusters is seen in the picture. On the basis of shape of the curved dislocations in the lower half area, the marked arrow (S) indicates the possible direction of the stress. More details of this picture will be explained in the discussion.

As mentioned before in section IIB-2, the observed voids are fewer in the thicker areas of the sample because of a less "effective thickness contrast". This is apparent from the Fig. 6 where the void density seems to decrease in the regions away from the perforation boundary in the lower left part of this figure.

Figure 9 shows some interesting shapes of voids which are somewhat flatter forms of the voids. One such shape is seen in the Fig. 6 also. This may be a stage in the life of the void before its collapse into the dislocation loop as postulated by Kuhlmann-Wilsdorf and Wilsdorf\textsuperscript{14}. These voids seem to have grown very large before such a possible collapse into loops. The larger void appears to
Fig. 8: Dislocation Motion in the Field of Vacancy Clusters
\[ \text{lcm} = 1560\text{Å}^0 \]

Fig. 9: Flatter Forms of Voids
\[ \text{lcm} = 1070\text{Å}^0 \]
be a normal stable form of truncated octahedron.

The sizes of voids cannot be measured accurately. By a rough measurement, an average size of the voids in Fig. 6 is about 200-250 Å and in the Fig. 9 is about 550 Å which may be the largest size of a thermal void in aluminum reported so far in the literature. In most cases only the central region of the sliced samples was examined for voids. A few samples were examined in the edge-region of the sliced disc-shaped specimens by repolishing to enlarge the initial perforation. A lesser density of voids compared to dislocation loops was observed in this region. Because this area has a steeper slope leaving a very narrow electron transparent region, no definite conclusions can be drawn. In an attempt to observe the voids in the skin region of the samples, the etched and cleaned samples were wrapped in an etched and cleaned foil and heat treated for creating voids. In this foil a large number of dislocation loops were found entangled into the dislocation forests created by unwrapping the foil for polishing. It is felt that the same technique may be used more precisely with a flat specimen, but it was not attempted again.

B. The Strength and Deformation Characteristics

1. General

The second phase and the primary aim of this investigation was to find the nature of the void strengthening in
aluminum. Some general observations are made here before going into the details of the results.

Below ambient temperature, a very large variation of the ultimate tensile strength was observed from sample to sample. The variation in the yield strength was relatively smaller and where it was quite significant, it is shown by an error bar in the figures 10, 11 and 12. By assuming that half of the yield strength is shear strength, its correlation with a strengthening mechanism is better. Therefore only the yield strength measurements are used here.

Sometimes a marked inhomogeneous deformation of the gauge length occurred. This was observed as a bamboo structure in the deformed sample. A localized shear fracture occurred many times except at the lower temperatures. A larger inhomogeneous deformation was usually followed by a localized shear fracture.

Because of the occurrence of frequent inhomogeneous deformation, in larger and smaller areas, the crosshead speed does not really represent the strain rate. In the case of strengthening due to the presence of vacancy clusters, particularly in the neutron irradiated single crystals of copper, Schwink and Neuhauser have shown that the effective gauge length varies with the crosshead speed so the strain rate as well as the yield strength is affected. It must be pointed out here that the analysis
of the present results becomes extremely difficult if the above complications are to be resolved. Hence, for simplicity in presenting these results and in spite of the possible criticism, the crosshead speed is assumed to represent the strain rate.

2. Variation of yield strength with testing temperature

Figure 10 shows the temperature dependence of yield strength. The curves have been drawn using the rationalized yield strength. For this rationalization, the values of G at various temperatures were taken from those given by Sutton\textsuperscript{63} for the elastic constant C\textsubscript{44}. The void-strengthened samples were tested at two strain rates, 3.33 x 10\textsuperscript{-2}/sec and 1.67 x 10\textsuperscript{-3}/sec while the well annealed samples, tested for comparison, were tested over the temperature range at a strain rate of 1.67 x 10\textsuperscript{-3}/sec and at room temperature and at 77\textdegree K using a strain rate of 3.33 x 10\textsuperscript{-2}/sec. The annealed samples were held in air at 600\textdegree C for 6 hours and furnace cooled (temperature and time is same as that used for the pretreatment of the void strengthened samples). In general, the void strengthened samples show a large temperature dependence of yield strength but its nature changes in five different temperature intervals from 77\textdegree K to about 600\textdegree K. A narrow temperature region with a width of about 10\textdegree K near room temperature divides the whole range into two regions,
Fig. 10: Temperature Dependence of Yield Strength
with dissimilar behavior below and similar behavior above room temperature. The broader transition zone at 413° + 20°K separates the region of linear temperature dependence of the yield strength at higher temperatures from the region of non-linear temperature dependence of the yield strength at the lower temperature. The well annealed samples have a much lesser temperature dependence of yield strength. They also appear to have a transition zone at about 413°K. The following observations are made from these curves about the void-strength samples.

a. Below the room temperature, the void-strength samples have shown a strange and unexpected type of temperature dependence of yield strength. The value of yield strength passes through a maximum or a minimum value depending on the strain rate. However, the yield strength at 77°K has not increased above that at room temperature.

b. At room temperature, an increase in yield strength to about 2.5 times the yield strength of well annealed samples has been obtained. This comparison changes considerably in a narrow temperature range just above the room temperature. At the higher strain rate, 3.33 x 10⁻²/sec, the variation of yield strength was quite uncertain because of a broad spectrum of
values at and near the room temperature. Small variations of temperature may have large effects as one would expect from the slope of the dashed curve. This uncertainty is shown by the dashed part of the curve.

c. Above room temperature up to about 393°K the yield strength of the void strengthened samples at the higher strain rate drops rapidly with increasing temperature in the beginning, then its decrease slows down at both the strain rates. It rises again and drops back in the next high temperature region of less than 40°K width which this author has termed as a transition zone, after which a linear decrease in the yield strength occurs with increasing temperature.

d. In the transition zone mentioned above at 413 + 20°K the rise and fall of the yield strength is very marked at the high strain rate, 3.33 x 10⁻²/sec. At this strain rate, the yield strength increased sharply to a maximum at about 413°K and then, although decreasing sharply, it remains quite high up to about 500°K.

At the lower strain rate, 1.67 x 10⁻³/sec, the transition zone is not so well marked. At this strain rate two of the six specimens tested
at 393°K showed repeated yield points on the load-elongation curve (the upper one corresponding to the values indicated in the curve in Fig. 10). One of the six specimens tested at 433°K showed a very sharp increase in the load after a large yield point elongation. This was considered as indicative of a possible change in hardening mechanism in the transition zone, since the well annealed samples tested at this strain rate, $1.67 \times 10^{-3}$/sec, showed a drop in yield strength in this temperature range.

e. Above approximately 450°K and up to the maximum testing temperature used here (600°K) the yield strength drops linearly with increasing temperature, at a faster rate at the higher strain rate.

3. The strain rate dependence of the yield strength,

The figures 11 and 12 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 was obtained for the well annealed samples as shown in the figures.

An empirical relationship between a flow stress and strain rate, at a constant temperature and strain, is
Fig. 11: Strain Rate Dependence of Yield Strength at 298°K
Fig. 12: Strain Rate Dependence of Yield Strength at 77°K
given by

\[ \sigma = C \left[ (\varepsilon')^m \right] \varepsilon T \]

where the coefficient \( m \) is called the strain rate sensitivity and \( C \) is an experimental constant. For the well annealed samples, the strain rate sensitivity, \( m \), is found to be approximately 0.042 at room temperature and 0.033 at 77°K. For all of these samples the sensitivity seemed much decreased at lower strain rates. In comparison the void-strengthened samples showed 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 has decreased at a higher strain rate of about 3.33 \( x 10^{-2} \)/sec, whereas at 77°K it has decreased at a lower strain rate, (3.33 \( x 10^{-5} \)/sec.).

In Fig. 13, the above data is plotted as the increment in the strain rate dependent yield strength of void strengthened samples above that of the annealed samples. The two curves corresponding to room temperature and to 77°K have a strongly inflected S-shape and the increment in the strain rate dependent yield strength tends to flatten at the lower and the higher strain rates.

C. Instability of Plastic Flow at Lower Temperatures

Many void-strengthened samples tested at liquid nitrogen temperature at the strain rate 3.33 \( x 10^{-4} \)/sec
Fig. 13: Increment in Strain Rate Dependent Yield Strength Above That of Annealed Samples
showed an unstable deformation characteristic wherein the load on the specimen dropped sharply accompanied by an easily audible click and further extension followed elastically until at least the original load was reached. This gave a serrated load-elongation curve as described by Basinski\(^6^4\) which did not have a uniform pattern. Since this behavior is unusual a load elongation curve showing such serrations is included in Appendix E. In one case, the load drop occurred within a fraction of one percent of the strain and in another, it occurred after a much longer extension, say 5 or 10 percent. The extent of the load drops ranged from a fraction of a percent of the load (prior to the click) to about 20% at times. This deformation characteristic was not observed in the lower and higher range of strain rates, but the possibility is not ruled out that this effect may have occurred on a much smaller scale.

The well annealed samples tested over the same range of strain rates at liquid N\(_2\) temperature did not show the above mentioned characteristics. A possibility of the formation of mechanical twins in the void-strengthened aluminum was ruled out because of the high stacking fault energy of pure aluminum and also because such an instability of plastic flow of aluminum was observed by Basinski\(^6^4\) in tests at liquid helium temperature. No twinning was found metallographically. Basinski, in his
tests on aluminum of similar purity and similar diameter of the test specimens observed no drop of the load before about 8% elongation and the stress-strain curve of the aluminum at temperatures above the boiling point of helium was always completely smooth.
V. DISCUSSION

A. The Model

Makin et al.\textsuperscript{58} have observed a consistent relationship between the increased critical shear stress in neutron irradiated copper crystals and the density of black dot defects (vacancy clusters below the 50 Å size and considered different from the dislocation loops). However, a similar consistent relationship was found by them to be absent when considering the strength in relation with the larger vacancy clusters, i.e. the dislocation loops present in their samples. Westmacott\textsuperscript{32} found that the value of the increased shear stress was not related to the size and distribution of the observed dislocation loops in the quenched aluminum or during the process of annealing out of the loops. These two studies have clearly shown that the presence of the different types of vacancy clusters other than the observable dislocation loops is causing the marked strengthening in the neutron irradiated copper and the quenched aluminum respectively.

More direct evidence of a similar effect is found in the Fig. 8. The motion of dislocations does not seem to be obstructed by the presence of dislocation loops except by the loop marked R. A closer observation shows that the dislocations have bowed between some pinning points marked A to H in the central lower half of the picture. The
pinning points are seen as either the breaks or the hollow points in the dislocation lines. These points are thought to be very tiny voids. This shows that in comparison to the loops the voids are much more effective obstacles to the motion of dislocations and so in the presence of both, the voids may be predominantly responsible for the observed strengthening effect. In the central region D-H-E, the pinning points are either tiny voids or dislocation junctions where the broad dislocation lines seemed to have bowed between the closely spaced pinning points.

To distinguish as to whether the primary cause of strengthening was either the void-strengthening or loop-strengthening in such experiments, the temperature dependence of the yield strength variation, if really distinct, deserves more serious consideration than the quantitative agreement between the defect density and the resultant stress increase. However, the available theories, viz. for void-strengthening by the Coulomb\textsuperscript{6} or the Fleischer\textsuperscript{33,34} theory for the strengthening caused by tetragonal distortions, have been discovered to be misleading in this respect. The incorrect deductions of the temperature dependence of yield strength from the Coulomb theory and the Fleischer theory have followed not because the analysis was incorrect but because the model was inadequate or deficient in that respect. This is discussed in appendix A.
Therefore these theoretical predictions are not to be taken too seriously nor can the exact nature of the temperature dependence of the yield strength in case of void-strengthening and loop-strengthening be considered to be well established. This also removes the basis for the inference by Westmacott\textsuperscript{32} that the strengthening in the quenched aluminum samples is caused by clusters of vacancies thought to be ungrown nuclei consisting of 6-7 vacancies in a collapsed configuration. Since loop nucleation and void nucleation are fairly fast processes occurring within a few seconds after quenching (Fig. 1b), doubt arises as to why it takes a much longer time of a few hours after the quenching to develop the higher strength. This indicates that those vacancy clusters responsible for the strengthening are somewhat larger than the ungrown loop nuclei or it requires the vacancies to form certain other vacancy clusters to be effective obstacles to the dislocation motion. Secondly, in such a case, the escape of dislocations from the defects having atomic dimensions is expected to be associated with only a small activation energy. Hence a plausible mechanism of void-strengthening should be able to remove these deficiencies observed in the Coulomb and Fleischer theories as applied to the present investigation. The Ashby\textsuperscript{44} improvement on the Orowan stress when viewed from the Coulomb approach modifies the Coulomb escape energy value (and the energy criteria) to correct only two of his flaws mentioned in
the section IIC-1 of the Literature Review. The equation (1) given therein has been obtained from the Ashby treatment of the Orowan stress as given below and this will facilitate its interpretation as the Coulomb approach. The above discussed deficiencies disappear when the result of the merging of Coulomb's approach and the Orowan stress concept are combined with the results of Weeks et al. from the interaction energy concept. This follows later.

A dislocation pinned by a chain of voids spaced at an average distance, \( L \), along the dislocation line is shown in the Fig. 14, which is slightly modified for the case of voids from that of Ashby. During the process of dislocation bypassing between the voids by the applied force, \( F \), the dislocation is shown to remain in contact with but tangential to the surface of the voids.

Now assuming the energy per unit length of dislocation, \( E \), to be independent of the orientation of the dislocation segment, one can regard the dislocation line tension as \( T = E \), so the force exerted to move the dislocation into the shown bowed shape can be written as,

\[
F = 2 E \cos \theta.
\]

Substituting for the dislocation energy,

\[
E = A \left( \frac{G b^2}{4 \pi} \right) \ln \left( \frac{R}{r_0} \right)
\]

we obtain,

\[
F = A \left( \frac{G b^2}{2 \pi} \right) \cdot \cos \theta \cdot \ln \left( \frac{R}{r_0} \right)
\] (3)
Fig. 14: A Dislocation Pinned By a Chain of Voids
Where, angle $\theta$ represents the progress of bypassing of the dislocation segment.

$$R = \text{Outer cut off radius}, \quad \text{and}$$
$$r_o = \text{Inner cut off radius}.$$  

$A$ has the same meaning as in equation (1) of Section IIC-1. The plot of the applied force, $F$, given by equation (1) and the progress of bypassing given by angle $\theta$ is a Cos $\theta$ curve having the maximum at $\theta = 0^\circ$, (i.e. the formation of dislocation dipoles at the voids) and the maximum force is the force necessary for bypassing the dislocation between the voids.

However, considering now the interaction between the adjacent bowed segments from the same void, the interaction has the effect of lowering the energy, $E$, by an amount which depends on the separation between the two segments and varies with angle, $\theta$ and distance $C$ from the voids. The separation is given by $x(1+C \sin \theta)$ which replaces $R$ in equation (3); so the interaction energy is given by

$$E = A \left( \frac{Gb^2}{4\pi} \right) \ln \frac{x(1+C \sin \theta)}{r_o}$$

Using the boundary condition that when $\theta = 90^\circ$, the outer cut off radius $R = L$, we have $C = (L/x - 1)$ where $x =$ void diameter. Therefore,

$$E = A \left( \frac{Gb^2}{4\pi} \right) \ln \frac{x[1+(L/x - 1) \sin \theta]}{r_o}.$$
Or the applied force related to the progress of bypassing of dislocation between the voids is

\[ F = A \frac{GB^2}{2\pi} \cdot \cos \theta \ln \frac{x[1+(L/x - 1)\sin \theta]}{r_o} \]  

(4)

which has a maximum value at \( \theta \approx 15^\circ \) to \( 30^\circ \) for most values of \( x \) and \( L \) as determined by graphical methods.

This criterion for bypassing the dislocation between the chain of voids is similar to that of Coulomb and corresponds to forcing the dislocation into such a critical shape (represented by angle \( \theta \) for \( F_{\text{max}} \)) that further bowing and snapping of the dislocation from the pinning voids occurs by its own reaction between the adjacent segments.

The applied force can be related to the local shear stress by the relation

\[ \tau = \frac{F}{L} \]

where \( \tau = \text{force per unit length of the dislocation for the bypassing} \). Therefore,

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

(5)

where \( \tau = \text{applied shear stress} \). Since \( L/x \gg x/r_o \), a reasonable approximation of equation (5) is given by

\[ \tau = A \cdot \frac{Gb}{2\pi L} \cdot \cos \theta \cdot \ln \frac{L}{r_o} \sin \theta \]
which evaluated for $\theta \approx 20^\circ$ gives,

$$\tau = A \frac{G b}{2\pi} \frac{0.94}{L} \cdot \ln \left(\frac{0.34L}{r_0}\right)$$  \hspace{1cm} (6)

This is the same relation mentioned as equation (1).

Using this equation with the value of $L$ given by relation 2 (Section IIC-2) the expected increase in the yield strength due to the presence of an assumed homogeneous distribution of uniformly sized spherical voids is shown in Fig. 15. According to the 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. By increasing the void size alone, the increase in yield strength should be restricted by the consideration that only a certain maximum average size of the voids with the void density above the necessary minimum value can be accommodated in the volume of metal without forming a defect (such as a macrocrack or a microcrack) that affects the strength adversely. This is not clearly visible from the simple form of model given as equation (1) of this thesis. In practice, when the density of voids increases, the increase in the average void size is limited by the available vacancy supersaturation.

Allowing for inhomogeneous distribution of voids the observed void density is in the range of $10^{13}$-$10^{14}$ voids/cm$^3$ with the average void size about 100 A$^0$ diameter.

For this the predicted increase in the yield strength from
Fig. 15: Expected Increase in Yield Strength as a Function of Void Size and Void Density
Fig. 15 is about 1.6 Kg/mm$^2$ while the measured value of the same at room temperature is about 1.32 to 1.64 Kg/mm$^2$ above the yield strength of the annealed samples at corresponding strain rates. Thus the results are in a fairly good quantitative agreement with the expectations.

Other consequences of the model are:

a. The dislocation segment snaps away from the voids by its own reaction after bowing further to its critical shape; for this the only condition is that the voids need be just big 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. The numerical value of the attractive interaction energy between the void and the dislocation is thus unimportant and so the extent of void-strengthening could be considered independent of the void size distribution. This aspect is well brought out by the studies of Makin et al.$^{58}$. They concluded that although the vacancy clusters (black dot defects) responsible for the hardening in their neutron irradiated copper have different sizes up to a maximum of 50 Å, they all have approximately the same $F_{\text{max}}$, i.e. the average strength contribution.
b. Accordingly, the smallest (limiting) size of the voids acting as effective obstacles to the dislocation motion can be found by equating the total input energy for bowing out of the dislocation with the maximum interaction energy for a void sitting symmetrically on a screw dislocation. The value of the latter is given by Weeks et al. as

$$E_{\text{max}} = -\frac{Gb^2}{2\pi} \cdot \left(\frac{\pi^2}{12} + \ln \frac{a}{r_o}\right)$$

Where, \(a\) = radius of void

The total input energy is found by integrating the curve (equation (4) of this section) of applied force vs the bypassing angle, \(\theta\), over its range \(\pi/2\) to 0 radians, i.e.,

$$E_{\text{to}} = \int_{\pi/2}^{0} \frac{A \cdot Gb^2}{2\pi} \cdot \cos \theta \ln \left[\frac{x[1+(\frac{L}{x}-1)\sin \theta]}{r_o}\right] \, d\theta$$

$$= A \frac{Gb^2}{2\pi} \left\{1 - \ln \left[\frac{r_o}{r_o-(\frac{L}{2a})} \frac{2a}{L-2a}\right]\right\} \tag{7}$$

Where \(x = 2a\), the void diameter.

Equating the two energies, the minimum void radius, \(a_{\text{min}}\), will be given by the relation

$$a_{\text{min}} = \frac{A \cdot \left[1 - \ln(\frac{L}{r_o-(\frac{L}{2a})})\right]^{2a_{\text{min}}/L-2a_{\text{min}}}}{\frac{\pi^2}{12} + \ln \frac{a_{\text{min}}}{r_o}} \tag{8}$$
This relation is not very convenient to work with since the voids within the core radius cannot be neglected. However, it shows that the limiting size of the voids as effective obstacles to the dislocation motion is again a function of void spacing or the void density.

Briefly, these deductions, a and b, show that the mechanism and the effectiveness of voids as obstacles to the motion of a dislocation is independent of size of voids above the limiting void size as governed by the prevailing void density which, in case of aluminum, should be above $10^{12}$ voids/cm$^3$ to obtain a measurable increase in the yield strength. Therefore the extent of void strengthening is primarily dependent on the void density and much less dependent on the void size. Therefore the expectations of the figure 15 must be viewed with considerable limitations and extrapolations cannot be justified.

B. Instability of Plastic Flow at Low Temperatures

The void strengthened aluminum samples were found to be much more susceptible to the instability of plastic flow at low temperatures as the phenomenon occurred in a higher temperature range with early occurrence during the elongation and occasional higher percentage of load drops when compared with that reported by Basinski$^{64}$. This increased susceptibility is attributed to the spurt like dislocation motion as a group of dislocations under stress
leaves one chain of voids and bumps into the next chain of voids thus produces a sudden change in the density of mobile dislocations. This instability of plastic flow has interfered considerably in obtaining a correct picture of the variation of yield strength with the testing temperature in the low temperature range.

The reason for not observing the instability of plastic flow at the strain rates below and above $3.33 \times 10^{-4}$/sec may be that at the lower strain rate excess heat accumulation is avoided and at a higher strain rate the inhomogeneous deformation is spread over a larger region thus generating less heat per unit volume per unit time. This heat effect, although always not so marked, may help the thermal activation of slip over a wider temperature range below room temperature. Thus, it will be virtually impossible to control the effective deformation temperature and the strain rate during discontinuous flow. This is partially seen in the occurrence of the strange temperature dependence of the yield strength observed below room temperature.

C. Nature: Strain Rate Sensitivity and the Temperature Dependence of Yield Strength

The nature of void strengthened aluminum as observed from the figures 10, 11 and 12 can be explained by the simultaneous occurrence of various reactions having interrelated influences. It is difficult, however, to offer
an explanation in quantitative terms. So the observable reactions are dealt with here in qualitative terms.

1. Strain rate sensitivity of yield strength

The dislocation spacings in a strained crystal are so close (as compared to the void sizes) that a simple model will be deceptive without considering the obvious repulsive reactions between dislocations when they bump into the already obstructed dislocations at the voids. The repulsive reaction of the incoming dislocation will tend to lower the stress required for bypassing the obstructed dislocation between the voids. The attractive interactions between the chain of voids and the incoming dislocations will be expected to cause a spurt like dislocation motion under the applied stress thus giving rise to undetectably small sudden changes in strain rates within small regions. From the Orowan's equation for strain rate,

\[ \dot{\varepsilon} = bNV \]

Where \( b \) = Burgers vector

\( N \) = density of mobile dislocations, and

\( \mathbf{V} \mathbf{B} \) = mean velocity of the dislocations.

Gilman\(^{65}\) considers its time derivative for the collective behavior of the moving dislocations,

\[ \ddot{\varepsilon} = b(N a + \mathbf{V} \dot{N}) \]  

(9)

Where \( a = \dot{\mathbf{V}} \) = acceleration of the dislocations, and

\( \dot{N} \) = production rate of mobile dislocations.
In the above equation the second term consisting of the change in the dislocation density will predominate in influencing the change in strain rate. A slightly different relation for the spurt like dislocation motion is given by Mecking and Lücke\textsuperscript{66},

\[ \dot{\varepsilon} = L b \dot{N} \]  

(10)

Where \( L \) = distance traveled by the dislocations made mobile in the time interval, \( dt \).

Here, the stress dependence of the strain rate is given by the production rate of the mobile dislocations under the assumption of \( L \) being approximately independent of stress.

Gilman\textsuperscript{65} has suggested that the velocity of a set of interacting parallel dislocations moving along a glide plane is independent of the concentration of dislocations and drops very sharply after reaching a certain concentration of moving dislocations.

Indirect evidence of such a collective and spurt like dislocation motion occurring in the present tensile testing may be found in the following two observations.

a. By increase in the density of mobile dislocations and the accompanying bumping effect which lowers the stress required for the dislocation bypassing, the increase in the strength beyond yield point to the tensile strength is not observed.
b. Figures 11 and 12 show that the yield strength of the void strengthened samples compared to that of annealed samples has a higher strain rate sensitivity both at room temperature and at 77°K and its temperature variation is relatively smaller. The higher strain rate sensitivity is expected from the strain rate changes given by equations 9 and 10. Variation in the production rate of mobile dislocations in case of spurt like dislocation motion between the chain of voids will be much less affected by the temperature change from room temperature to 77°K. The almost constant difference (≈ 0.06) observed (Section IVB-3) between the strain rate sensitivity of yield strength of the void strengthened samples and the annealed samples at both room temperature and at 77°K is significant in this respect.

2. The temperature dependence of yield stress

The flow stress of this model given by

$$\tau = A \frac{Gb}{2\pi} \frac{0.94}{L} \ln \frac{0.34L}{r_o}$$

shows that the variation of yield strength under constant speed of testing should arise through:

a. the change in the average void spacing, L, by change in the void size and void density.
b. dissociation of dislocations during the dislocation movements which lowers the Burger's vector by an unknown amount.

c. the ease and the number of operative slip systems during the process of elongation at that testing temperature.

In practice, these considerations need to be extended to include the susceptibility of samples to the instability of plastic flow at lower temperatures together with the inhomogeneous deformation causing high local strain rates at times. Similarly at higher temperature some of the motion of dislocations will be thermally activated. However, when dislocation climb becomes operative at high temperature, an increase in the effectiveness of voids as obstacles to the dislocation motion can occur because the attractive interactions between the voids and the moving dislocations can prevent some of the dislocation climb.

Now it is possible to explain the observed variation of the yield strength (Fig. 10) of the void strengthened samples in the different temperature ranges described earlier. The yield strength of the samples tested at two strain rates varies strangely in the below ambient temperature regions. The rationalized ordinate in this figure is slightly lower at liquid nitrogen temperature than at room temperature. Normally the yield strength should rise with
the fall in testing temperature and the corresponding rise will be more for the test at higher strain rate. However, due to the inefficient heat transfer at low temperature due to decrease in specific heat and thermal conductivity, during elongation the accumulated heat in sample can cause the instability of plastic flow (sometimes audible) observed at 77°K. This will reduce the yield strength considerably. Also the sudden increase in the mobile dislocation density as the group of dislocations breaks away from a chain of voids to bump on to the dislocations in the next chain of voids, will lower the yield strength.

Thus, otherwise normally increasing yield strength with decreasing temperature and increasing strain rate is decreased in this below ambient temperature range in the following way:

a. assuming that the effect of the spurt like dislocation motion on the yield strength is independent of the temperature, but increasing with increasing strain rate and increasing stress value (i.e., with increased dislocation density).

b. the heat accumulation in samples due to inefficient heat transfer will be increasing with increasing strain rate. As mentioned before this may not cause the audible
instability of plastic flow if the deformation is spread over a larger volume, but it will provide thermal activation of dislocation motion. This heat accumulation will be lower at higher testing temperatures because of the improved thermal conductivity.

Combination of these two reasons is considered to be responsible for the observed dissimilar and strange temperature dependence of yield strength at the two strain rates below ambient temperatures.

In the temperature range near and above room temperature some thermal activation of dislocation motion and the spurt like dislocation motion will be expected to remain effective. The former will show increasing effectiveness in continuously lowering the yield strength of the void strengthened samples as the testing temperature is increased. The thermal activation effect is predominant in the temperature range above room temperature before any other mechanism can start operating.

In the temperature range 120°C to 160°C the yield strength at the strain rate 1.67 x 10^{-3}/sec changes negligibly, but a fast rise of yield strength occurs at the higher strain rate of 3.33 x 10^{-2}/sec. In case of well annealed samples a small drop, although not so certain, is found in the yield strength in this temperature range.
This gave rise to speculation that a change of work hardening mechanism is occurring over this temperature range. A slight variation in the load-elongation curve of only a few samples was also observed at 120°C and 160°C.

To explain the rise in yield strength in this temperature range at higher strain rate, it is suggested here that the climb of dislocations becomes operative from around 120°C and therefore with increasing temperature the voids become more effective in capturing the moving dislocations at the higher strain rate. However, this increasing effectiveness of voids toward strengthening will be nullified beyond a certain temperature by the increased thermal activation of the dislocation motion and the yield strength starts falling again. At the lower strain rate the increasing capture of moving dislocations will be less as the number of moving dislocations will be less in the same time since the simultaneously occurring thermal activation can nullify it or dominate the results to show continuously decreasing yield strength.

In the temperature range 160°C to 300°C the thermal activation of dislocation motion is rapidly increasing accompanied by an increase in the active slip systems and the resultant softening of material follows. Yet by the same considerations the strength of void strengthened samples is expected to be always greater or equal to that of the annealed samples which in this higher temperature
range show no hindrance to climb.

D. Shape of the Strain Rate Sensitivity Curve

The higher strain rate sensitivity of void-strengthened aluminum has been discussed earlier in terms of the behavior of dislocation motion between the chain of voids, viz., spurt like dislocation motion. Independent of this, an additional consideration is provided here for this same phenomenon.

The shape of the strain rate sensitivity curves in Figure 12 and Figure 13 will be of some interest in context with the theory proposed by Hart\textsuperscript{67} for the flow of polycrystalline materials. According to his theory, the general shape of the strain rate sensitivity curve for the polycrystalline or multiphase specimen is expected to fall into a strongly inflected S curve as shown in Figure 16. The curve would normally extend over a very wide range of strain rates. As evidence he has shown a similar curve from the data of Cline and Alden\textsuperscript{67} for Sn-Pb eutectic alloy for which the curve is compressed into a smaller range of strain rates. He then explains the shape of the curve on the basis of a model in which the steady state deformation of a polycrystalline material is regarded as the flow of a non-Newtonian body, the matrix, which contains a dispersion of plate like flaws (grain boundaries) that permit shear sliding at their surfaces.
Fig. 16: Shape of Strain Rate Dependent Yield Strength

Fig. 17: Increment in Stress Due to Change in Strain Rate Sensitivity
governed by Newtonian friction.

The similar shape of the curve, assuming it to be spread over a larger range of strain rates than those used in this thesis, will not be visible for the well annealed polycrystalline samples tested. However, the polycrystalline void-strengthened samples have shown a shape representing the available range of strain rates used for the normal tensile testing used. This leads to the suspicion that the extra strain rate sensitivity has arisen by the distribution of voids (added to the distribution of plate like flaws, grain boundaries) in the non-Newtonian matrix. If so, a similar shape of the curve will also be found in single crystals of the void-strengthened samples, but spread over a relatively larger range of strain rates. However, if the range of strain rates remains unchanged from that in the present studies, the formerly described mechanism, i.e., spurt like dislocation motion, may be considered primarily responsible for the observed rise of the strain rate sensitivity in the present case.
VI. CONCLUSIONS

1. The model presented here for the void strengthening directly follows from the Orowan stress with Ashby's improved criterion. The same when viewed from the Coulomb point of view offers a more satisfactory mechanism for void strengthening. The increased yield strength at room temperature obtained in the void strengthened aluminum samples agrees fairly well with the predicted values on the basis of this model.

2. The mechanism of void strengthening and the effectiveness of voids as obstacles to the dislocation motion and therefore the extent of void strengthening are primarily dependent on the void density and much less dependent on the void size. The above interpretation of this model has been arrived at by a logical extension of the available information, i.e., by combining the Orowan stress concept in the model with the interaction energy concept of Weeks et al. 45

3. The void strengthened aluminum samples were found to be much more susceptible to the instability of plastic flow at low temperatures. This increased susceptibility is attributed to the spurt like dislocation motion as a group of dislocations under
stress leaves one chain of voids to bump into an other chain of voids, gives rise to sudden changes in strain rate and inhomogeneous deformation in the microregions.

4. The temperature dependence of yield strength in void strengthening as well as in loop strengthening predicted in literature is argued to be deceptive.

The observed temperature dependence of yield strength in the void strengthened aluminum is strongly affected by different reactions occurring concurrently and hence, it follows a changing pattern in different temperature ranges. The yield strength is found to be highly temperature dependent near room temperature decreasing rapidly with rise in temperature.

5. There is a possible change in the work hardening mechanism of aluminum between the temperature $120^\circ C$ and $160^\circ C$ during which the void strengthened aluminum samples show much less temperature dependence of the yield strength. Also the effectiveness of voids as obstacles to dislocation motion is more striking in this temperature range.

Under various competing mechanisms the void strengthened aluminum retains higher strength than the annealed samples over a wide temperature range.
6. The yield strength of the void strengthened aluminum samples compared to that of the annealed aluminum is found to be more sensitive to the strain rate both at room temperature and at 77°K. This increased strain rate sensitivity of yield strength of the void strengthened samples is attributed both to the spurt like dislocation motion and to the voids acting as additional flaws to permit shear sliding at their surfaces by Newtonian friction as postulated by Hart.
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VITA

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APPENDIX A

THE TEMPERATURE DEPENDENCE OF YIELD STRESS
IN THE COULOMB AND THE FLEISCHER THEORIES

A. The Coulomb Theory of Void Strengthening

The escape energy for dislocations to break away from
the chain of voids is given by Coulomb\textsuperscript{6} as

\[ \Delta E = Gb^2 r \cdot \alpha (1 - \frac{2\beta L}{b} \varepsilon) \]  \hspace{1cm} (A1)

This has been substituted for the activation energy
in the strain rate relationship, as given by Coulomb,

\[ \frac{d\varepsilon}{dt} = \frac{nvb^2 N L D^2}{(2L)^2} e^{-\frac{\Delta E}{kT}} \]  \hspace{1cm} (A2)

to give the final relationship,

\[ \sigma = G\varepsilon = \frac{Gb}{2bL} \left[ 1 - \frac{kT}{Gb^2 \cdot r \cdot \alpha} \log \frac{nvb^2 N L D^2}{(2L)^2 \frac{d\varepsilon}{dt}} \right] \]  \hspace{1cm} (A3)

where \( \alpha \) and \( \beta \) are Coulomb's constants.

\[ \frac{d\varepsilon}{dt} = \text{Strain rate in tension} \]

\( N = \text{Dislocation density present in the sample} \)

\( L = \text{Total length of dislocations per unit volume of the sample} \)

\( r = \text{Radius of the voids} \)

\( L = \text{Spacing between the voids along the dislocation length} \)
From the equations (1) and (3) it is clear that if
\[
\frac{n v b^2 NLD^2}{(21)^2}
\]
is considered constant, by increasing the testing temperature in tests at constant strain rate the quantity \(\Delta E/kT\) must remain constant, i.e. by increasing the temperature the \(\Delta E\) given by the equation (A1) will increase and therefore the strain, \(\varepsilon\), required to dislodge the dislocation from the voids should decrease with increasing temperature. This relation, then, expresses greater sensitivity of the stress with the change in testing temperature since the temperature is in the denominator of \(-\Delta E/kT\) which is a power of the exponential. Secondly, amongst the multiplying factors preceding the exponential the atomic frequency, \(v\), will increase slightly with temperature so the corresponding change in \(\Delta E\) should be even more than that expressed by \(\Delta E/kT = \text{constant at the constant strain rate.}\)

This could be verified experimentally from equation (A3) by testing the void-strengthened samples at two different strain rates at constant temperature and solving the two equations simultaneously. Unless the measured stresses are almost equal at the different strain rates, the slope of the curve, stress vs. temperature,
$\Delta \sigma /^{0}K$ will be expected to be quite high for aluminum and other metals. This is contrary to the Coulomb demonstration of the expected results.

B. The Fleischer Theory of Solution Hardening by Tetragonal Distortions

The temperature dependence of the shear stress of Fleischer's model\textsuperscript{33,34} arises from the force-distance curves calculated by him for the tetragonal distortions acting as obstacles to the dislocation motion in F.C.C. crystals. This curve is an equilateral hyperbola for which he finds the force, $F$, on the dislocation as a function of distance, $x$, along the slip plane for a region $x > b$,

$$F \propto F_0 \left( \frac{x}{b} + 1 \right)^{-2} \quad (A4)$$

Where $F_0 = G\Delta \epsilon b^2 / 3.86$ and $\Delta \epsilon = \text{difference between longitudinal and transverse strain of the tetragonal distortion}$.

Therefore the activation energy for the slip by the applied force, $F$, is found to be

$$U = F_0 b \left[ 1 - \left( \frac{t}{t_0} \right)^{1/2} \right]^2 \quad (A5)$$

which is equated to $\alpha kT$ since for the dislocation to move at a fixed velocity (a given number of jumps per second) the energy must be some multiple of $kT$, i.e., where

$\alpha = \text{Experimental constant}$

$k = \text{Boltzman's constant, and}$
$T = \text{Absolute temperature.}$

Thus equation (5) is expressed as

$$\left(\frac{T}{T_0}\right)^{1/2} = 1 - \left(\frac{T}{T_0}\right)^{1/2}$$

(A6)

where $T_0 = F_0 b / \alpha k$ and $\tau = \text{Shear stress.}$

The temperature dependence of the shear stress given by the above equation (A6) will be satisfied by any other type of interaction between the dislocation and the defects if the shape of their force-distance curve in the short range is similar to that used by Fleischer.

According to the more rigorous calculations of Weeks et al.\(^{45}\) for the normalized interaction energy as a function of the separation distance between a bubble and a screw dislocation, the force distance curve for the void-strengthening is more likely to be similar to that roughly derived by Fleischer in the range $x > b$ for the solution hardening by tetragonal distortions. Therefore the predicted temperature dependence of the shear stress given by the Fleischer theory cannot be made a criterion to distinguish between the loop strengthening and the void strengthening.
APPENDIX B

AN HYPOTHESIS ON FATIGUE FAILURE IN POLYCRYSTALS

This digression from the present investigation is prompted by its significance with regard to the fatigue life of components.

The strain rate sensitivity, \( m \), of a metal given by

\[
\frac{\Delta \log \text{ (yield stress)}}{\Delta \log \text{ (strain rate)}}
\]

changes continuously with changes in temperature, and below a certain temperature it increases with increasing temperature. The theory presented by Hart suggests that the strain rate sensitivity at constant temperature of a polycrystalline material will show different values in different ranges of strain rates. These two effects are shown schematically in Fig. 16 where the strain rate sensitivity at any strain rate is given by the slope of the curve.

If the increment between the strain rate dependent yield strength of a polycrystalline sample in the heat treated condition over that of a proper reference state, (say, the annealed state) is considered then the increment between the strengths at two fixed temperatures will be lower at the lower strain rate and probably the same will be true at very high strain rates. The reason is that the yield strength cannot be expected to keep rising continuously.
with increasing strain rates. The general shape of the curve showing the increment of the strain rate dependent yield strength with reference to the annealed state is again a strongly inflected S curve. By a proper choice of a reference state (for comparison) of the material the two isotherms may give a closed S shaped loop with the lower isotherm corresponding to a lower temperature, or, they may not cross each other depending on the temperature of these isotherms. This situation is shown in Fig. 17 (in Discussion). Without consideration for any particular state of the material this schematic figure shows two isotherms, each indicating the increment in the stress variation at that temperature due to a change in strain rate sensitivity (caused by the change in strain rate) corresponding to the plotted strain rate.

Now, if the stress related to the strain rate, i.e. the stress increment depending on strain rate, is considered the energy per unit volume, an interesting phenomenon may take place in a rotating shaft. As the shaft rotates the nature (tension or compression) and amount of strain changes in a cyclic form. The rate of change of strain rate in any region at any instant is governed by the angular velocity of the shaft and the amount of strain. In this cyclic process when the strain rate is increasing in elongation the stress (not necessarily the yield stress) in the region increases (for the same
amount of strain but with different strain rate sensitivity). This strain rate dependent stress increases along an isotherm and the stress falls off along the same isotherm when the strain rate is decreasing in tension. Similar stress variations with changing strain rates during the compression cycles will occur along an isotherm. Assuming that the energy input in the shaft in the elongation and compression (i.e. with strain) under load is completely reversible, these energy changes due to the strain rate changes are not reversible and will appear as heat created by friction within the shaft. The intermittent rise in temperature can shift the process to different isotherms but unless the heat is dissipated effectively the accumulated heat can activate slip and the temperature falls slightly. Numerous repetitions of this process aided by inefficient heat conduction will be detrimental to the fatigue life of the component with or without the above process being responsible for initiating a crack.

Thus the fatigue life of a component is related, according to this hypothesis, to the strain rate sensitivity of the material and the bulk thermal conductivity.

It is interesting to note that the strain rate dependent behavior of most polycrystalline metals follows in shape or nature the different regions of this hypothetical curve given by Fig. 17. The strain rate
sensitivity of many common metals (such as iron and aluminum) falls into the lower left part of the curve where the rate of change of strain rate sensitivity is lower in a lower temperature range and is higher in a higher temperature range. Some of the superplastic alloys (e.g. Zn-Al alloy) at higher temperatures fall into the higher right hand region of the curve while the curve shifts leftward on the strain rate axis. The results of the void strengthening of aluminum shown in Fig. 13 fall in the wide central region between the isotherms of Fig. 17. However the significance of this hypothetical curve is not clear.
APPENDIX C

VOID FORMATION: COMMENT ABOUT A POSSIBLE ROLE OF IMPURITY ATOMS AND A TWO STAGE NUCLEATION PROCESS

Although the formation of voids is the first important step in this investigation, the mechanism of void formation was not considered in detail. The preparatory studies have led to certain thinking which has been offered here. These ideas without concrete evidence to justify them are included here because they hint at a method to obtain a suitable void distribution in the mass of sample.

Shimomura and Yoshida\textsuperscript{28} have shown that hydrogen is required in the heat treating atmospheres for the formation of voids. Foster et al.\textsuperscript{68} have shown that radioactive hydrogen follows some impurity atoms (yet unknown) in the 99.9999% pure Al and therefore they have suggested that the migration of hydrogen atoms to certain impurity atoms at high temperature can form the "nuclei bubble", viz. a nucleus having enough gas pressure inside to support a surface, after cooling to room temperature.

Heterogeneous nucleation is always easier than homogeneous nucleation and there is more evidence to show that the void formation is heterogeneously nucleated. However, it looks more probable that void formation is a
two stage nucleation process suggested by Foster et al.\textsuperscript{68} instead of a single stage nucleation process involving vacancies moving to form clusters around hydrogen atoms. In the two stage nucleation process, the hydrogen atmosphere around the impurity atoms may be forming at higher temperatures which creates the suspicion that they could be hydride forming elements gathering excess hydrogen. The number of impurity atoms needed to give a void density of $10^{16}$ voids/cm$^3$ is so small that it may be detectable only for known impurity atoms.
APPENDIX D

SAMPLE CALCULATIONS FOR A TYPICAL RUN OF TENSILE TESTS

Gauge length = 1 inch, Cross head speed = 0.1 inch/min hence the strain = 1.67 x 10^{-3}/sec.

Chart speed = 20 inches/min, therefore magnification of the elongation axis of the load-elongation curve on the chart is 200 X. So the 0.1% proof stress is given by 0.2 inch along the elongation axis.

Average diameter of the samples is 3.13 mm, or the cross sectional area is 0.01195 sq. inch, so one pound of load on the sample represents a stress of 0.05872 kg/mm².

Measured loads at yield point of a batch of six void strengthened aluminum samples tested at room temperature, 298°K, are:

(1) 39 lbs, (2) 39 lbs, (3) 37 lbs, (4) Damaged, (5) 40 lbs and (6) 38 lbs.

The average value is 39 lbs which corresponds to an average yield strength of 2.29 Kg/mm². The minimum and the maximum yield loads of this batch, 37 lbs and 40 lbs respectively, correspond to the minimum and the maximum yield strength of 2.17 Kg/mm² and 2.35 Kg/mm² respectively.

Using the value of C_{44} at 298°K from Sutton as a value of shear modulus, G, (thus G = 2.78 x 10^{11} dynes/cm²)
the rationalized values \((\sigma y/G)\) of the average, minimum and maximum yield strength of the batch are \(8.09 \times 10^{-4}\), \(7.66 \times 10^{-4}\) and \(8.30 \times 10^{-4}\) respectively.

Such calculations have been used to find points plotted in the Figs. 10, 11 and 12.
A Serrated Stress-Strain Curve Showing Instability of Plastic Flow at Low Temperatures