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Reply to "Comment on Defects in Aluminum Quenched from the Liquid State"

By G. Thomas* and R. H. Willens†

K. A. Jackson[1] recently published some comments regarding our data on quenched liquid aluminum.[2] The latter data had been recompared to equilibrium data on vacancy concentrations in solid aluminum.[3] Jackson argued that our experimental results[2] should not have been corrected for denuded zones,[2] and that we had overestimated the vacancy concentrations. We had supposed that under the conditions of splat cooling, vacancies lying within ~1500A° of the freezing surfaces would have escaped, so that an effective foil thickness was used to measure the defect density. The object of this note is to prove that there is vacancy denudation at and near surfaces, and hence that our original measurements of vacancy concentrations in quenched liquid aluminum are correct or still possibly underestimated.

The results pertinent to our arguments are shown in figures 1-3, which are micrographs of aluminum after quenching from ~1000°C. These examples provide the following information:

1) Regions of foils thinner than about 1500-2000A° contain no resolvable dislocation loops nor small clusters even after many tilting experiments to provide differing contrast conditions both in bright and dark-fields (Figs. 1,2).

2) At wedge shaped crystals (e.g., at edges or near holes) no defects are visible within the first one to three extinction distances of the edges (Figs. 1(b), 3). Since the extinction distances for the reflections used

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are about 650A° for $\bar{g} = \langle 111 \rangle$ and about 750A° for $\bar{g} = \langle 200 \rangle$ it can concluded that very few or no defects exist in foils within about 1500A° of free surfaces. Thus when dealing with measurements of loop concentrations in "splat cooling" experiments the effective foil thickness to be used is about (t-1500)A°, where t is the actual thickness. This is the value we previously adopted.[2]

3) The denuded regions near grain boundaries exist to about the same extent as those near the free surfaces.

4) If dark-field images are taken under $s \neq o$ conditions (absorbing cases) the relative position of defects within the foil can be approximately ascertained (for details see ref. 4), and images can be formed separately of defects near either surface. Such an experiment is illustrated by Fig. 3.

   Since most of the defects appear for both $s>o$ and $s<o$, they must all lie about the same distance from either surface, that is, near the middle of the foil. Furthermore the intensity of an image from a defect near the foil surface can be estimated by the most intense grain boundary fringe. It can be seen from Fig. 3, that at $s \neq o$, very few defects have intensities comparable to that of the fringe, also indicating that there is a strongly denuded region near each surface. Also, defects near the surfaces would show strain contrast images, i.e., black-white lobe like images, for non-zero values of s, while defects in the interior would appear as simple spot-like diffraction images, as do most of the images observed.[5,6] These results strongly indicate that in splat cooled foils no vacancy loops exist near the surfaces.

We have re-estimated the vacancy concentrations in foils after quenching from 1000°C by taking measurements of defect densities near thickness fringes (see e.g., Figs. 1,3)- From the latter, t can be estimated to within at least a couple of hundred A°. The average defect concentration for quenches from 1000°C are at least $10^{16}/cm^3$, in complete agreement with our first published data.[2]

We have also looked carefully at the defect distribution in foils prepared by electropolishing quenched solid bulk specimens of aluminum. Results at and near the edges of foils are similar to those shown for splat cooled foils in Figs. 1 and 3. Thus even in foils prepared from quenched bulk crystals appreciable vacancy denudation occurs near free surfaces.

The manner by which vacancy depletion occurs at surfaces is of interest. Unclustered vacancies will be lost simply by migrating to the sinks during solidification or quenching. In addition loops once formed can also be lost either by gliding or climbing to the surface under the influence of image forces.[7] A rough calculation of the distance from the foil surfaces from which depletion of loops occurs due to the latter effect gives about 300A° from each surface. A further factor (not considered by Jackson) which must also be accounted for is the invisibility of loops due to the diffraction conditions. For example if the defects exist as Frank loops, all are visible for $\bar{g}$ = <200> but only one-half are visible for $\bar{g}$ = <220>; for perfect loops, one-third are invisible for $\bar{g}$ = <200> and one-sixth are invisible for $\bar{g}$ = <220>. Also, loops smaller than about 50A° are not readily resolved.[5] Thus determinations of vacancy concentrations based on measurements of loop density will usually tend to be underestimated, even from foils prepared from bulk material.

In view of the foregoing it is suggested that the following methods be adopted for measuring loop densities:

1) Obtain the thickness as accurately as possible, either by observing defects along constant thickness fringes or by forcing dislocations to glide to provide slip traces.

2) Because of vacancy losses near free surfaces, the effective thickness of the foil should be taken approximately as (t-1500)A° when computing the defect density. It is obvious that less error from this effect occurs if measurements are made from the thickest regions of foils. However if the foil is too thick it may be difficult to resolve defects near the center of the foil because of the effect of absorption on the intensity.

3) Ensure that as many defects as possible are made visible by taking photographs of the same area under different, known, diffracting conditions.

Jackson[1] also argues that the higher the pre-quench temperature, the slower is the cooling rate. This is contrary to experience in the preparation of metastable phases by the rapid quench technique. Usually, the alloy to be quenched is heated significantly higher than the melting point to reduce the viscosity of the liquid. With a lower viscosity, the bulk of the foil tends to spread thinner and hence quench faster.

To summarize, we have shown conclusively that vacancies are lost near free surfaces and that our original estimates of vacancy concentrations in splat cooled aluminum are, (Fig. 1, ref. 3) if anything still underestimated, and that Jackson's assumptions[1] are not justified.

ACKNOWLEDGEMENTS

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FIGURE CAPTIONS

Figure 1(a) Aluminum splat cooled from ~1050°C; general view showing absence of dislocation loops near free surfaces E, grain boundaries G and holes H. Bright-field image.

Figure 1(b) Same area as (a) tilted to show wedge fringes, operating reflection [111]. Notice that no loops can be seen within the first three extinction fringes from the edge i.e., for foil thicknesses less than ~2000A°.

Figure 2 As Fig. 1, showing absence of loops in thin areas and near grain boundaries (tilted to show thickness fringes at boundaries) bright-field image.

Figure 3 Aluminum splat quenched from ~1000°C

(a) bright-field (b) dark-field $s>o$ (c) dark-field $s\approx o$

(d) dark-field $s<o$

Most of the defects are visible under all three dark-field conditions. Very few defects are visible within the first 1500A° thickness from the edge.

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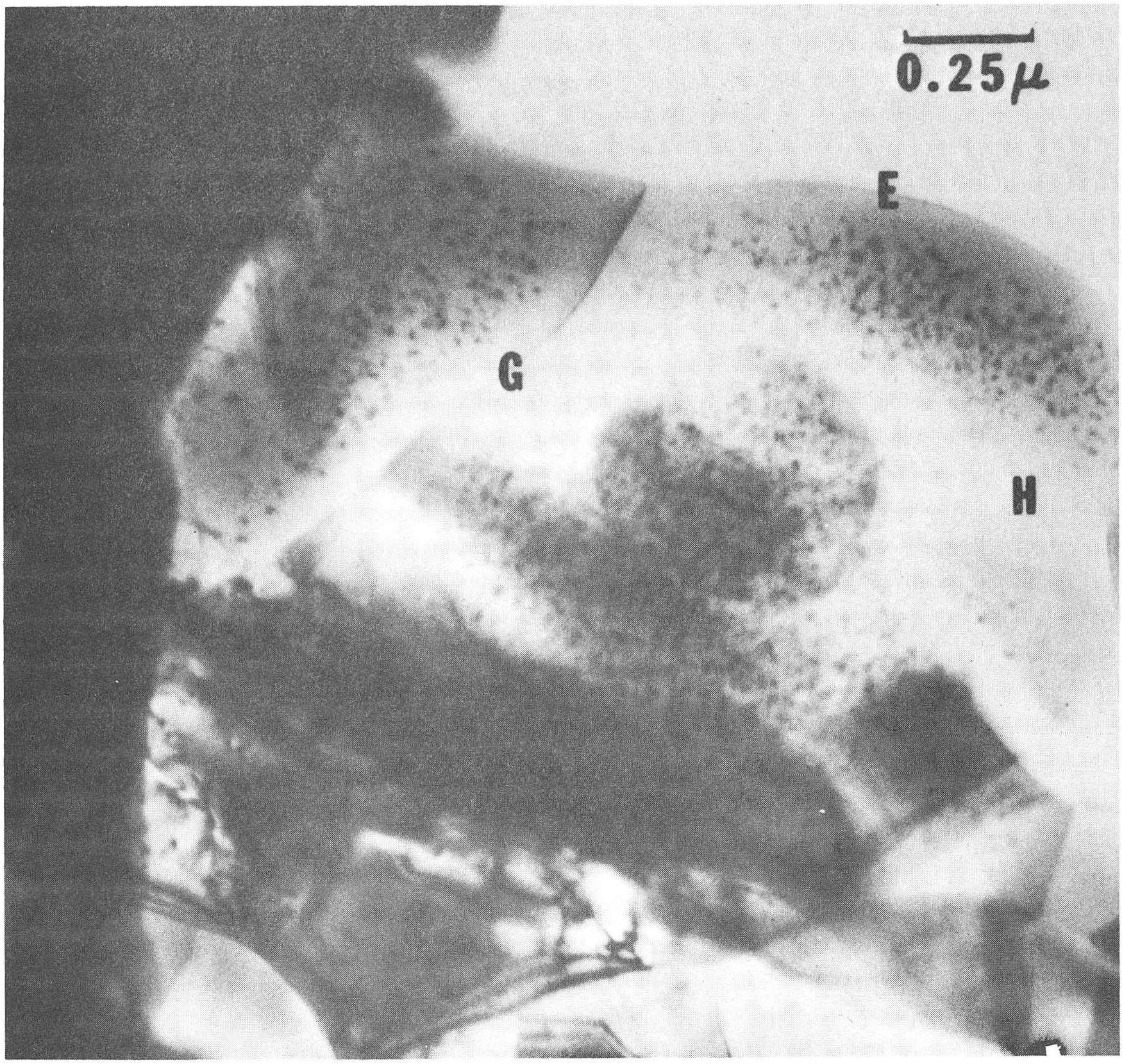
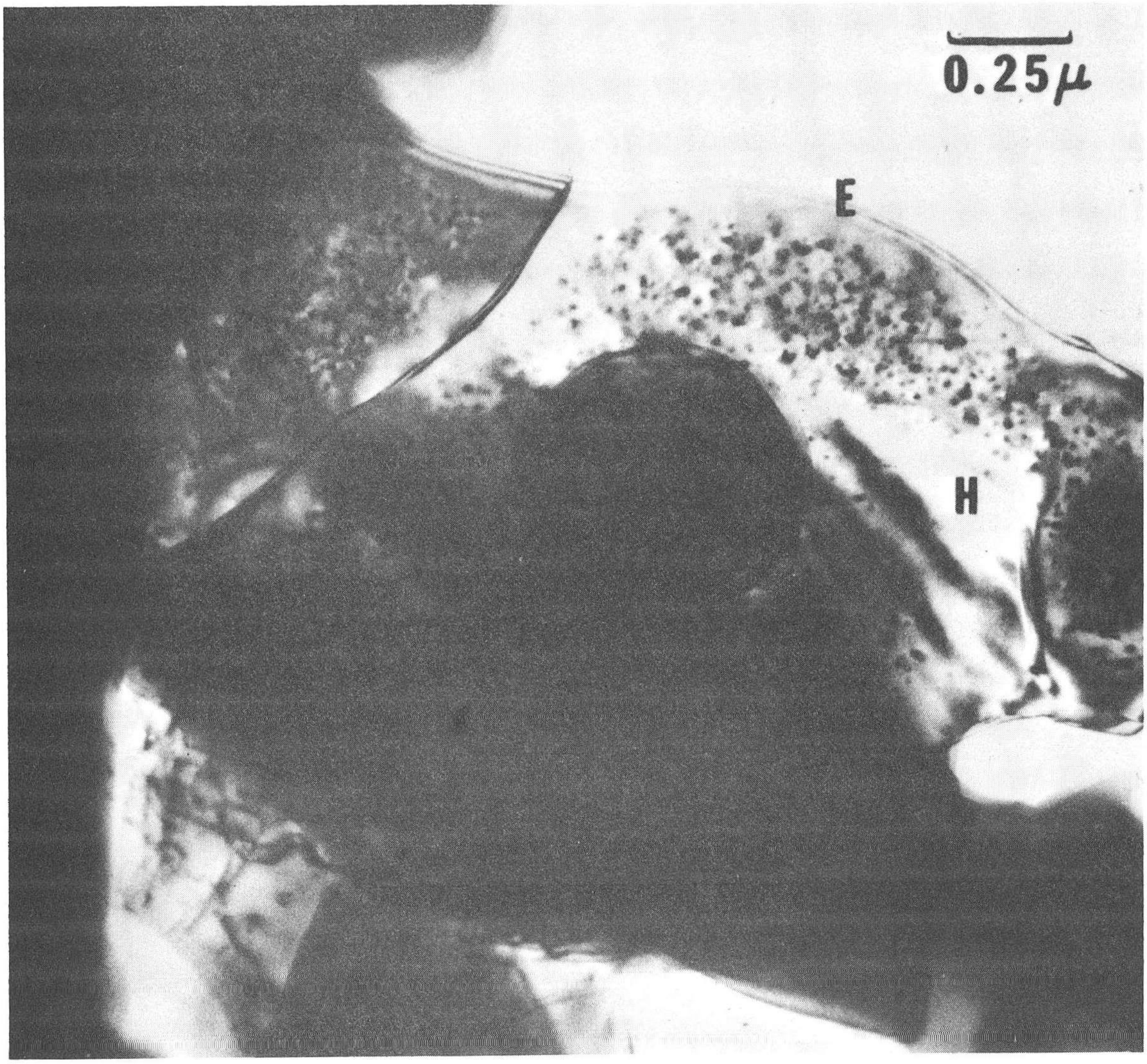


ZN-5491

Fig. 1a



ZN-5492

Fig. 1b

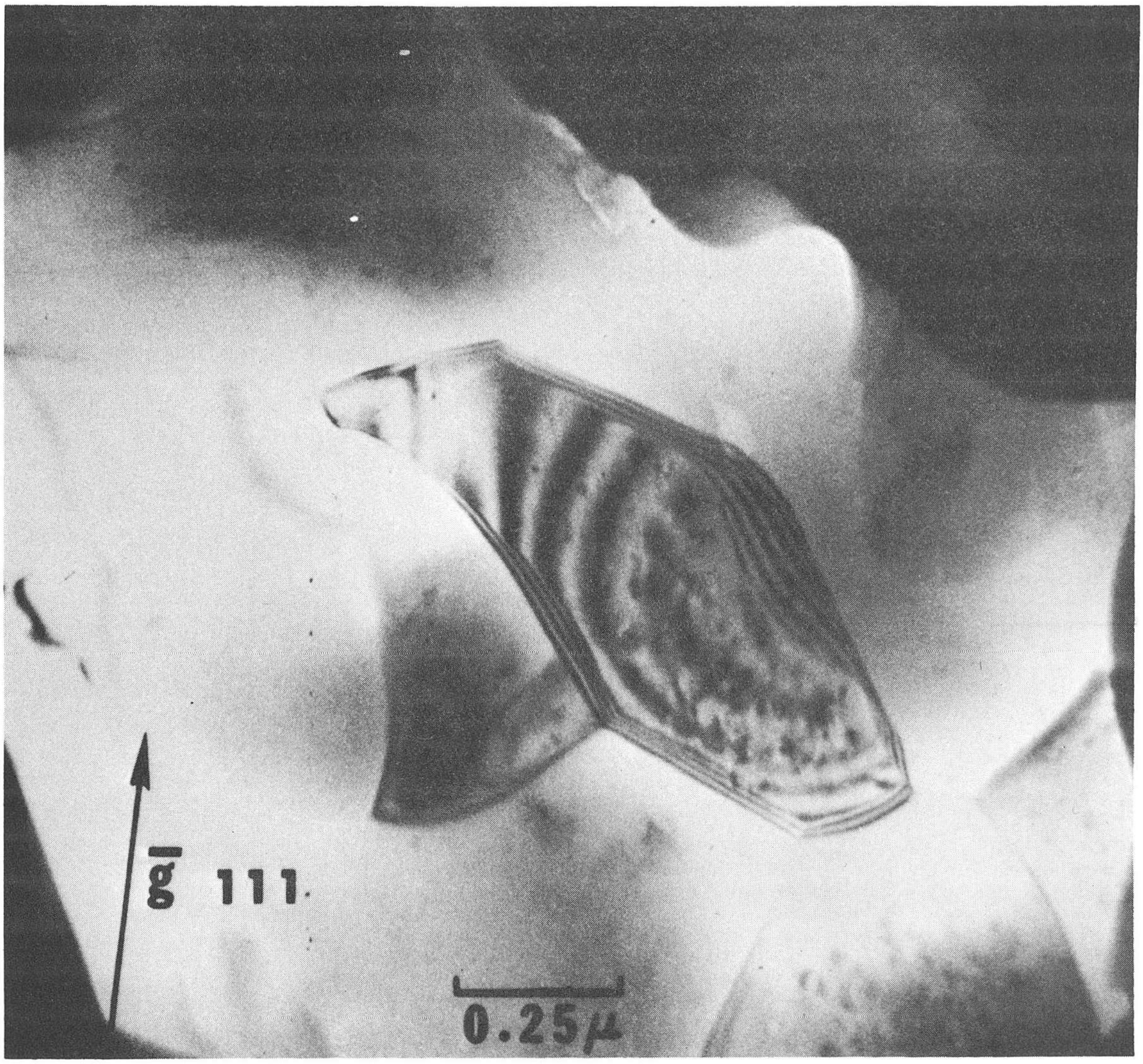


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Fig. 2

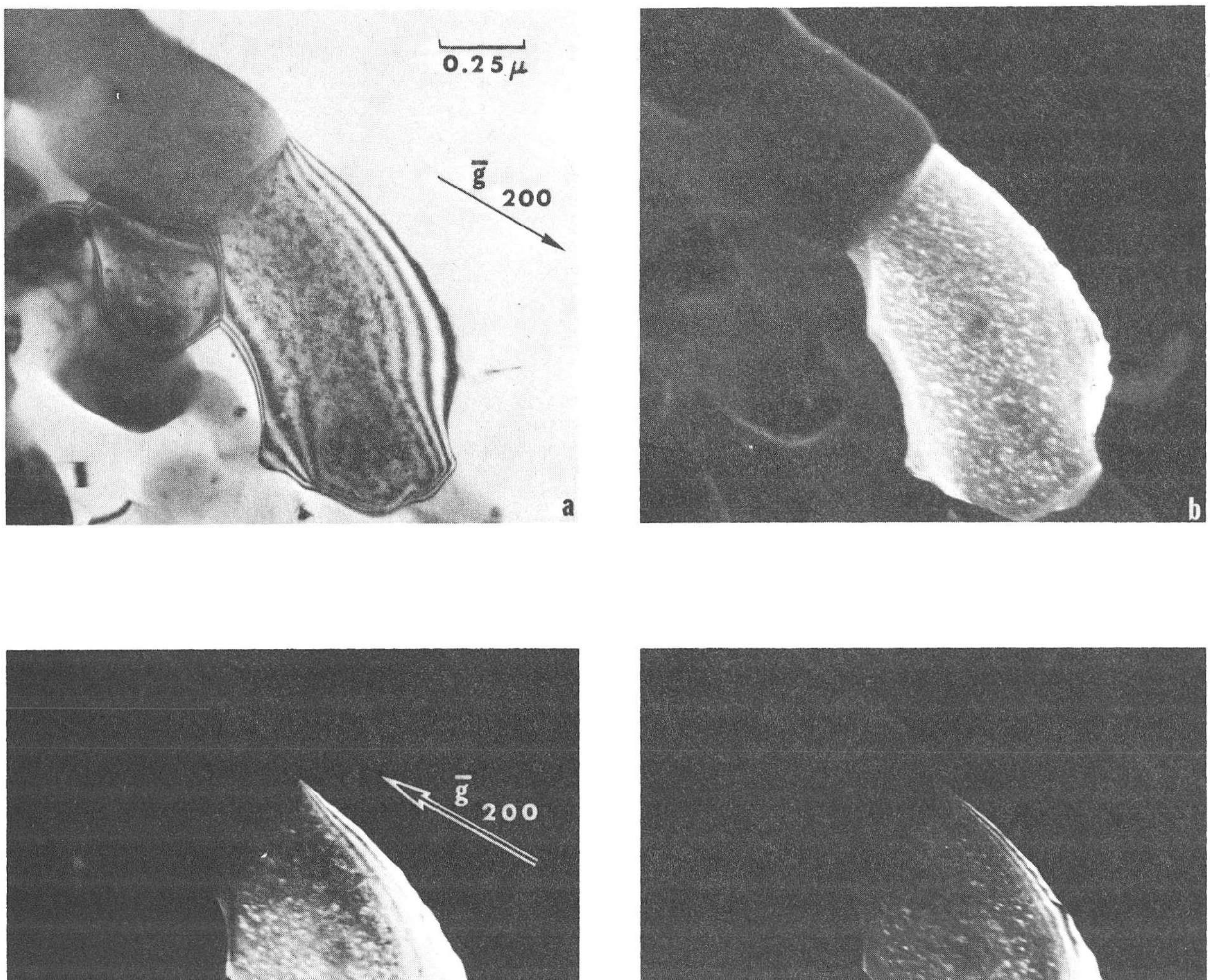

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Fig. 3