is localized precipitation on the dislocation loop, the standard test is to "extinguish" the dislocation contrast; the dislocation will remain visible if it has fine precipitates because of their extra contrast.

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Precision Density Measurements as a Tool for the Investigation of Point Defects

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The technique of measuring smallest density changes of macroscopic metal specimens, based on Archimedes principle, has been improved to the extent that relative density changes as small as 3.5×10^{-7} can be detected. At the same time the experimental effort involved has been reduced. A further improvement, to a sensitivity of about 2×10^{-7}, is believed possible with only little additional effort. The method shall be employed for an investigation into the production or absorption of point defects and their aggregates by moving dislocations, and also into the annealing behavior of these defects. A preliminary result is in accord with a previous prediction, namely that annealed f.c.c. metals contain vacancies condensed into submicroscopic voids, and that these are eliminated by moving dislocations, leading to an initial increase of density in specimens deformed after annealing. The density increase in coarse grained aluminum specimens, deformed in tension by about 2%, is tentatively given as $\Delta\rho/\rho=(1.32\pm0.09)\times10^{-5}$.

1. Introduction

The most direct measuring technique by which the behavior of point defects in general and of lattice vacancies in particular may be investigated, presumably lies in sensitive determination of density changes[1)-5)]. Unfortunately, with previously available methods the best resolution obtainable lay in the order of $\Delta\rho/\rho=10^{-5}$ or at most 5×10^{-6} for the atomic fraction of vacancies. This sensitivity is unsatisfactory since the total fraction of thermal vacancies in a solid usually is below 10^{-4}, and since even a fraction of 10^{-6} vacancies is believed to give rise to significant effects. For this reason an improved technique for the measurement of small density changes in macroscopic solid specimens has been devised. It is a further development of the method described by Bell[6)], in which the weight difference between a specimen and a dummy of the same material and of closely similar mass and dimensions is determined, first in air and then in a suitable liquid.

2. Experimental Technique

For the case that specimen and dummy have closely similar mass, that the specimen is subject to a treatment which changes its density but not its mass, and that the density of the air is neglected compared with the density of the weighing liquid, the relative density change of the specimen is given by $\Delta\rho/\rho=\Delta W_L/V\rho_L$, where V is the specimen volume, ρ_L is the density of the liquid. ΔW_L is the change of the measured weight difference between specimen and dummy, when both are in the weighing liquid, before and after the treatment. Consequently, the smallest relative density change which can be measured in a specimen of constant mass is limited by the smallest detectable weight difference $\delta(\Delta W_L)$ when specimen and dummy are compared while immersed in the weighing liquid. This in turn is almost always limited by unavoidable temperature fluctuations. Let the temperature difference between the liquid immediately surrounding the specimen and that surrounding the dummy be ΔT, then the resultant difference in buoyancy is $V(\alpha_L-\alpha_S)\rho_L\Delta T=\Delta W_T$, where α_L and α_S are the coefficients of thermal volume expansion of liquid and specimen material. Thus the smallest density change that can be measured in the presence of uncontrolled temperature differences of magnitude ΔT is given by $\delta(\Delta\rho/\rho)=\delta(\Delta W_T)/V\rho_T=(\alpha_L-\alpha_S)\Delta T$. Typically, $\rho_L=1$g/cc, and $(\alpha_L-\alpha_S)$ between $5\times10^{-4}/°C$ and $10^{-3}/°C$. It appears that ΔT can rarely be reduced much below $10^{-2}°C$ so that $\delta(\Delta\rho/\rho)$ at best becomes 5 to 10×10^{-6}.

Previously, in order to reduce ΔT to as low a level as possible, stringent measures of temperature control were taken, such as controlling the temperature of the balance room as well as of the liquid bath, in which both specimen and control specimen were immersed. Moreover, the liquid bath was often made quite voluminous and was constantly stirred.

A drastic improvement of sensitivity, however may be achieved by a different approach as follows:

1) The container for the liquid bath is made thick-walled, out of solid pure aluminum, or copper or other suitable metal, with U-shaped cross section such that it fits into the balance case. Hereby the wide arms of the U receive the specimen and the dummy, and the somewhat narrower bottom of the U passes behind the main post of the balance. In this way, it is assured that the liquid level is the same for dummy and specimen, and convection of the liquid may take place so as to even out temperature differences. Heat conduction through the walls of the metal vessel reduces any local temperature differences still further.

2) No additional measures for temperature control are taken, neither in the balance room as such nor with respect to the weighing liquid. It so happens that the balance room used in the experiments described is situated in a basement, away from outer walls, so that for this reason conditions are particularly favorable. On the other hand, temperature control of the balance room was originally attempted until it was found that it introduced, rather than eliminated, temperature fluctuations in the liquid bath.

3) The temperature difference between the dummy and the specimen is measured and the weighings corrected appropriately. To this end, two similar, slotted copper cylinders, made by bending rectangular pieces of 1mm thick copper sheet into a cylindrical shape, are immersed into the liquid such that the specimen and the dummy are each surrounded by one of them without touching. A short constantan wire, the two ends of which are soldered to the two copper cylinders, serves to establish a differential thermocouple of copper-constantan-copper. Two copper leads, one each soldered to one of the cylinders, and connected to a sensitive galvanometer, complete the circuit for measuring the thermo-voltage, generated through temperature differences between specimen and dummy. This electrical circuit is insulated from the aluminum container through a lining of glass plates.

All in all, points (1) to (3) above, represent a very substantial reduction in experimental effort compared to earlier methods. Moreover, weighings can be taken without lengthy waiting periods as soon as the liquid has come to rest, say, 20 minutes after inserting the specimens. These advantages are particularly gratifying because the described method, when coupled with sound weighing techniques, renders a very high sensitivity.

3. Discussion of Results to Evaluate the Technique

So far, three weighing sequences have been completed, namely two with a coil of coarse grained aluminum wire of 10 g mass, one before and one after slight straining, and a third sequence with a solid aluminum cylinder of 50 g mass. The sensitivity of the Ainsworth semimicro balance of type TX was adjusted to 3.3 divisions/10^{-5} g weight when used with the 10 g specimen, and the sensitivity of the thermo-voltage circuit amounted to $3.9_3\times10^{-3}°C$/mm deflection of the galvano meter light spot. With the known coefficients of thermal expansion, namely $\alpha_L=8.78\times10^{-4}/°C$ for diethylphthalate and $\alpha_{Al}=0.72\times10^{-4}/°C$ for aluminum, the correction factor thus was 4.34 divisions/mm for the case of the coil specimens. Since with 50 g specimens

Table I

B Reading of Balance, in Divisions	B_0 Zero Point of Balance, in Divisions	$B-B_0$ Measured Weight Difference, in Divisions	ΔT Galvanometer Reading, in mm	$\Delta B=4.34 \times \Delta T$, Temperature Correction, in Divisions	$B_{corr}= B-B_0+\Delta B$, Corrected Balance Reading, in Divisions	δ Deviation from Mean Corrected Balance Reading of 21.2 div., in Divisions	δ^2
$\left.\begin{matrix}39\\36\\34\\37\end{matrix}\right\}$ 36.5	−14	22.5	0	0	22.5	+1.3	1.69
$\left.\begin{matrix}-2\\1\end{matrix}\right\}$ −0.5	−19	18.5	+0.25	+1.1	19.6	−1.6	2.56
$\left.\begin{matrix}10\\19\end{matrix}\right\}$ 14.5	− 7	21.5	0	0	21.5	+0.3	0.09
13	− 7	20	+0.5	+2.2	22.2	+1.0	1.00
2	−10	12	+2.0	+8.7	20.7	−0.5	0.25
19	− 5	24	−1.0	−4.3	19.7	−1.5	2.25
11	− 4	15	+1.75	+7.6	22.6	+1.4	1.96

$\bar{B}_{corr}$=Mean weight difference, corrected for temperature: 21.2 div. $\sum\delta^2=9.80$ div².

Legend: Complete record of measurements taken on undeformed coil of aluminum wire of 10 *g* mass, weighed against comparison specimen. Weighing liquid was diethylphthalate. Horizontal lines indicate that specimen and dummy were cleaned and dried before reweighing.

the sensitivity of the balance was only 3.0_5 divisions/10^{-5} g the correction factor for the 50 g specimen amounted to 20.0_5 divisions/mm.

Table I gives the complete sequence of weighings with the coil specimen before straining. In this table, a horizontal line indicates that specimen and dummy were removed from the balance together with their suspension hooks, were rinsed in alcohol and ether and were dried, before inserting them again into the balance. Thus the table represents seven discrete weighings, extending altogether over several days. The galvanometer could be read to 0.5mm, and estimates to 1/4mm were attempted. From these measurements, the probable error for each weighing may be found as $(\sum\delta^2/n\text{-}1)^{1/2}=1.28$ divisions $=3.9\times10^{-6}$g, corresponding to $\delta(\Delta T)=0.3$mm$=1.16\times10^{-3}$°C.

For the complete sequence of seven weighings, the probable error (i) interpreted as error in the weighing is $\delta(\Delta W_L)=0.48$ divisions $=1.46\times10^{-6}$g, (ii) interpreted as error in the temperature measurement is $\delta(\Delta T)=0.112$mm $=4.4\times10^{-4}$°C, and (iii) for the measurement of relative density change is $\delta(\Delta\rho/\rho)=1.46\times10^{-6}/3.70\times1.118=3.5\times10^{-7}$ (The volume of the coils is 3.70cc and the density of the liquid at 20°C is 1.118g/cc).

The other two sequences of measurements gave quite similar probable errors for $\delta(\Delta T)$ and for $\delta(\Delta\rho/\rho)$, with $\delta(\Delta\rho/\rho)=4.5\times10^{-7}$ a conservative estimate for the average probable error of relative density measurement. On the other hand, the scatter of the measurements as interpreted in terms of *weighing* error is much larger for the big specimens than for the coils. From this result it is clear that the limiting factor at present is the accuracy with which the temperature difference between the specimen and the dummy can be read. This conclusion is consistent with the fact that the probable error in each temperature measurement was found to be $\delta(\Delta T)=0.3$mm, (see table I), since it involves two readings (actual reading and zero point reading) each estimated at best to the nearest 1/4mm.

Further information is to be gained from a plot of all measured values of $(B-B_0)-\bar{B}_{corr}$ as defined in Table 1 (except for two which were believed to be in error) *versus* the measured temperature difference ΔT, (Fig. 1). In addition to the measured points, the theoretical curves for the 10 g and 50 g specimens have been entered as solid lines.

Evidently, the scatter of the points for the two different specimens is quite similar when interpreted as errors in temperature reading, but are widely different in terms of errors in weighing. This is, of course, nothing but a graphical representation of the above conclusion that the limiting factor at present is lack of sensitivity in the galvanometer used.

Secondly, the scatter of the points is apparently quite independent of ΔT, the value of the temperature difference between the specimen and the dummy. In other words, temperature-corrected measurements obtained from weighings taken when the temperature was nearly uniform were no more reliable than when the temperature difference was comparatively large. Hence convection currents do not add to the error in any noticeable measure.

Thirdly, the average value of ΔT in all measurements was about 8×10^{-3}°C, corresponding to $\delta(\Delta\rho/\rho)\simeq6\times10^{-6}$, *i.e.* the temperature uniformity achieved by the simple means described is not at all inferior to the best obtained with rigorous methods of temperature control, judging from the highest sensitivities claimed in earlier investigations[1-6].

4. Application of Technique

As stated above, the method was developed with the aim of investigating the production and elimination of point defect aggregates by dislocations, as well as the annealing behavior of these crystal defects. One of the specific problems of considerable interest relates to the predicted existence of sub-

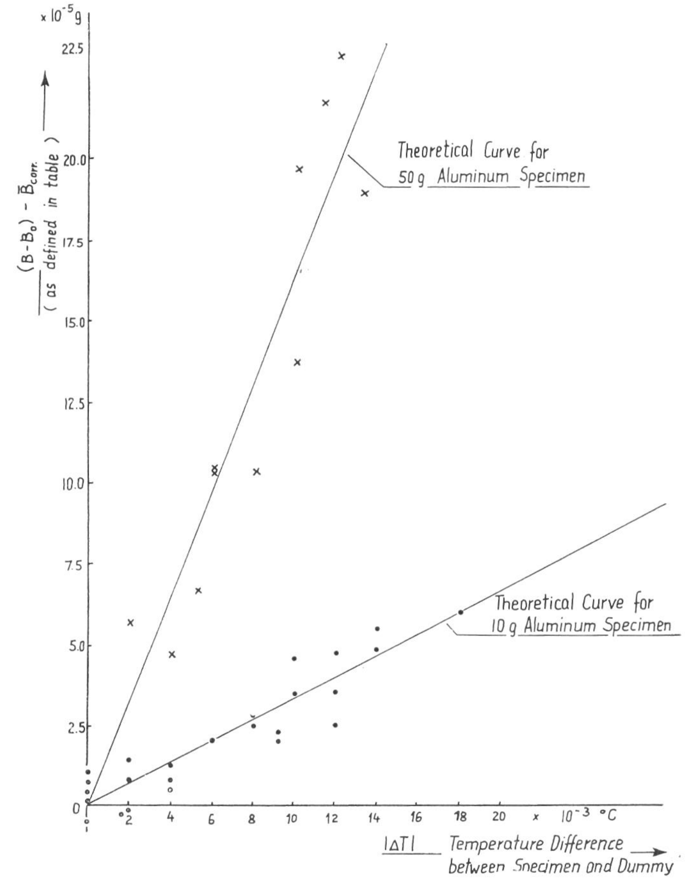

Fig. 1. A plot of measured values of $(\bar{B}-B_0)-\bar{B}_{\rm corr}$ as defined in Table I, *versus* ΔT, the measured magnitude of the temperature difference between specimen and dummy. The two solid lines indicate the theoretical curves for 10 g and 50 g specimens.

microscopic voids, previously beyond the reach of experimental investigation. It had been deduced from indirect evidence[7),8)] that such voids exist in annealed f.c.c. metals and that they are eliminated through the action of glide dislocations, thereby giving rise to dislocation tangling even at very small strains. Consequently it was expected that the density of annealed coarse-grained aluminum would rise when it was slightly strained.

In order to test this hypothesis the specimens in this investigation were given the form of cylindrical coils of wires with a bamboo structure. The advantage of this specimen shape is that a uniform widening of the diameter of the coil can be effected without undesirable grip effects, and that it dose result in a tensile strain. Had the specimens been compressed or rolled, then a density increase due to the expected effect could not have been distinguished from a trivial effect, namely the mechanical closing up of smallest gas-filled cavities.

The preliminary result of this investigation was that a tensile strain of about 2% resulted in a density increase of $\Delta\rho/\rho=(1.32\pm0.09)\times10^{-5}$. This is a rather large effect, well beyond the probable error of about 6×10^{-7} due to combining the results of two sequences of weighings. As a matter of fact, the sensitivity of the balance itself is only known within approximately 2%, increasing the overall uncertainty of the density change measured to about the limits stated. Still, it seems most desirable to repeat the experiment at least twice before placing definite reliance on the result.

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DISCUSSION

Simmons, R. O.: First question: How do you propose to avoid the problems of possible macroscopic flaws, voids, microcracks, and other inhomogeneities? Such problems might be suspected to appear after plastic deformation, and be difficult to devise precautions against using density measurements alone. Second question: Density measurements would appear to be less direct than energy release studies, for example. Because the important part of the information desired relates to defect concentrations, how do you obtain such concentrations directly from density changes alone?

Kuhlmann-Wilsdorf, D.: (i) In order to avoid the possibility that the closing of cracks or macroscopic voids may mask the looked-for effect, the present experiments were performed in tension; as explained in the written report on this lecture. Further, sequences of weighings after annealing treatments can, of course, be utilized to distinguish point defect behavior from trivial effects, and to identify the defects. This, however, is the same for other methods too. (ii) We, as well as other investigators, will doubtlessly find it advantageous on occasion to combine density measurements with other experimental techniques. I do not know of any technique which by itself is capable of absolute determinations of point defect concentrations, assuming that neither the specific volume, nor the specific electrical resistivity, nor the point defect energy, nor any other such quantities are known. In this respect, density determinations do not differ from other techniques.