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THE STACKING FAULT ENERGY IN THE Ag-In SERIES

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ABSTRACT

The intrinsic stacking fault energy has been determined in the Ag-In series using extended nodes, from pure silver to Ag-12.5 wt.% In (e/a = 1.23). Extrinsic faulting has been observed throughout the series, and examples are shown both of extrinsic-intrinsic node pairs, and the extrinsic-intrinsic fault pairs reported earlier by Gallagher. The present results show that the stacking fault energy is insensitive to alloying up to e/a ~ 1.04, after which it decreases with increasing solute content. Both for high solute content alloys and for practically pure silver, the extrinsic and intrinsic stacking fault energies are approximately equal. Following high temperature annealing treatments the effective stacking fault energy in high solute content alloys is observed to increase irreversibly, suggesting the presence of a solute impedance force at room temperature.

I. INTRODUCTION

The importance of the stacking fault energy (γ) in f.c.c. elements and alloys is such that it is desirable to know the manner in which γ varies with alloying. Adler and Wagner (1962) using x-rays examined a number of silver alloy series including that investigated in the present work, and obtained the faulting probability as a function of solute. Vassamillet and Massalski (1963) investigated several series using x-ray scattering and placed their results on an absolute scale by fitting their points at high solute content to those of Howie and Swann (1961). The latter workers examined extended nodes in copper and silver alloys, and nickel cobalt, and linearly extrapolated their results at low solute content in order to estimate the stacking fault energy of the pure metals. The validity of such an extrapolation was placed in some doubt, however, by the work with copper alloys of Peissker (1965), who, from extensive measurements of τ_3, the stress at the beginning of stage 3, as a function of strain rate, found a peak in the plot of γ against solute content at an electron-atom ratio, $e/a = 1.04$.

Fortunately, Loretto et al. (1964) and Gallagher (1964) have shown that even in pure silver extended nodes may be observed and measured, so that γ may be determined from node measurements as a function of solute content from $e/a = 1.00$ to the phase boundary. Because of the uncertainty which exists over the dependence of γ on solute content in dilute alloys particular emphasis has been placed on this region in the present work. An examination of dilute alloys is also of interest since it can give an indication of the likely effects of the segregation of impurities in the pure metal.

It is felt that the most recent theories relating extended node parameters to the stacking fault energy (Brown, 1964; Siems, 1964; Jøssang et al., 1965) can be used with confidence, since they have been shown to predict well the observed experimental configurations (Gallagher, 1966a). A further advantage of determining the stacking fault energy from direct observation in the electron microscope is that a distinction can be readily made between intrinsic and extrinsic faulting. It has been shown recently that extrinsic faulting occurs in low stacking fault energy materials (Loretto, 1964; Ives and Ruff, 1966; Gallagher, 1966b) and in the present work extrinsic faults have also been observed in samples whose stacking fault energy is ~20 ergs/cm^2.

II. EXPERIMENTAL PROCEDURE

The silver samples were prepared from deoxidized material of 99.9999% purity, and 99.999% purity indium was used in the alloys which were prepared in evacuated Vycor capsules. After quenching the ingots were cold-rolled to .007" thickness, and homogenized at 500°C for 48 hours under a vacuum of 5.10^{-6} torr. Spectrographic analysis of each of the alloys showed impurity contents less than 10 p.p.m. After light deformation in torsion the samples were thinned electrolytically (dilute alloys and pure silver, 6 gm KCN/100 cc H_2O, potential 2.5 v; other alloys, 70% ethyl alcohol, 20% perchloric acid, 10% glycerin, -20°C, 10 v).

To facilitate the measurement of the inscribed radius (w) of the small nodes which are observed in silver and its dilute alloys subsidiary negatives were prepared at a further magnification of x17.5 from the electron microscope plated (x20,000). When the subsidiary negative was

viewed with a further magnification of x4 in a Vanguard Motion Analyzer, the inscribed radius of the smallest nodes had been magnified to ~1 cm and could be readily measured. When measuring such small nodes (w ~ 85Å) care must be taken to ensure that systematic errors do not arise from the diffracting conditions.

Measurements of w are generally made under imaging conditions which yield stacking fault contrast. In the present work, the rolling texture leads to a [110] foil normal, and suitable reflections are 002, $1\bar{1}1$ or $\bar{1}11$. Nodes may be observed in either the $11\bar{1}$ or 111 planes and for any of the above reflections one of the total dislocations which form the node and two of the partials which bound it are out of contrast. The partial dislocation which is in contrast lies opposite the total dislocation which is out of contrast.

For small nodes, particularly, it is important to consider the above facts when making measurements of the inscribed radius. Howie and Whelan (1962) have used the dynamical theory to determine the intensity distribution of the image about the partial dislocation which separates a stacking fault from perfect crystal. Their results show that when the partial dislocation is in contrast ($\underline{g} \cdot \underline{b} = \pm 2/3$) the position of the partial corresponds closely to that point at which the intensity characteristic of the faulted region starts to decrease from its maximum value. However, when the partial is out of contrast ($\underline{g} \cdot \underline{b} = \pm 1/3$) the position of the partial correlates closely with the point at which the intensity characteristic of the faulted region has decreased to its minimum value. That the theoretical predictions agree well with observations of stacking faults

taken under different diffracting conditions was shown by Gallagher (1966b) [Figure 10].

Attempts have been made to measure small nodes using a microdensitometer, but the method is time consuming, and because of the shape of the node the inherent accuracy of the instrument is difficult to achieve. Accurate results can be obtained by fitting a circle to a tracing of the node, the tracing having been made with subjective allowance for the imaging conditions. If the lines of the tracing are fitted to the node image along regions of identical intensity for all three partials, then the measurements either underestimate or overestimate the true node size.

If all three partials are treated as if $\underline{g} \cdot \underline{b} = 1/3$ one line of the tracing will lie a distance x outside its true position, where $x \sim 0.25\xi_g$ (Howie and Whelan, 1962) and ξ_g is the extinction distance for the operative reflection. The consequent overestimate in w, the inscribed radius, is $\sim 0.08\ \xi_g$, and ξ_g in silver for 111 and 002 reflections is ~200Å. By treating all three partials as if $\underline{g} \cdot \underline{b} = 2/3$ two lines of the tracing are in error, and the inscribed radius is underestimated by $\sim 0.16\xi_g$, leading to an error in w of about 30Å. Such errors must clearly be avoided when w is only ~85Å as in pure silver.

III. RESULTS

3.1 Intrinsic Stacking Fault Energy

Figure 1 illustrates typical nodes in each of the alloys which have been examined. The magnitude of w, the inscribed radius, varies by a factor of 5 from pure silver to the alloy with highest solute content.

To investigate the effect of oxygen on the stacking fault energy ingots of pure silver and of the alloy with e/a = 1.10 were prepared in air rather than under vacuum. Vacuum fusion analysis indicated that even this treatment had resulted in only a few parts per million of oxygen (~5 to 8 p.p.m.) being absorbed, but it is felt that particularly in alloys all the oxygen is not revealed by this procedure. Helium 3 activation analysis utilizing the reactions $O^{16}(He^3,p)F^{18}$ and $O^{16}(He^3,n)Ne^{18} \xrightarrow[1.5\ sec]{\beta^+} F^{18}$ after Markowitz and Hall (1959) is to be used by the present authors in order to determine the total amount of oxygen absorbed by the specimens.

Table 1 contains details of the measurements which have been made and the calculations therefrom. The values of $\bar{w}$ have been corrected for the inclination of the node in the foil, and the standard error of both a single reading and of the mean is shown. The value of ν and G, Poisson's ratio and the shear modulus respectively, are calculated from the data on the elastic constants in Ag-In by Bacon and Smith (1956), using the relationships between ν, G and the elastic constants from Aerts et al. (1962). In this way some allowance has been made for anisotropy since the values of ν and G are those appropriate to dislocations in {111} planes, and are exact for dislocations along <110> directions. The intrinsic stacking fault energy (γ) has been calculated using the equation relating w to γ given by Brown and Thölén (1964).

The striking feature of the results in Table 1 is the insensitivity of γ on e/a for the dilute alloys. The present results, in conjunction with those of Peissker (1965) indicate that a linear extrapolation from

a plot of γ against e/a does not lead to a correct determination of γ in the unalloyed metal. In contrast to the results of Peissker, however, in the Ag-In series (with alloying elements of the present purity and oxygen content) a peak is not observed in the value of γ for dilute alloys.

Figure 2 compares the results of Table 1 with the earlier work of Howie and Swann (1961). Their results have been re-calculated using the equations of Brown and Thölén (1964). Allowing for the fact that different solutes have been used, the curves are quite similar, the only major difference being that the points for the most dilute alloys in Ag-Al and Ag-Zn series lie somewhat above the Ag-In curve. The magnitude of the stacking fault energy for pure silver determined in the present work (γ_{Ag} = 21.9 ergs/cm^2) agrees well with the previous results of Loretto et al. (1964) (γ_{Ag}= 21 ± 7 ergs/cm^2) and Gallagher (1964) (γ_{Ag} = 20 ± 6 ergs/cm^2). Also shown in Fig. 2 are the results from extrinsic-intrinsic fault pairs (Gallagher, 1966b) and these will be discussed in section 3.2.

In the present work the effect of oxygen on the stacking fault energy in pure silver is small. From measurements on 10 nodes in specimens of pure silver which had been melted in air, the stacking fault energy was determined as 23.8 ± 1.5 ergs/cm^2, compared with γ = 21.9 ± 1 ergs/cm^2 in silver with a lower oxygen content. Using the τ_3 method to determine the stacking fault energy in de-oxidized silver, Ahlers (1965)found γ_{Ag} = 15 ± 6 ergs/cm^2 while in earlier work on silver with a higher oxygen content Ahlers and Haasen (1962)found γ_{Ag} = 65 ± 8 ergs/cm^2. Further discussion is speculative until more reliable quantitative

information on the oxygen content and distribution is available from the experiments mentioned earlier. However, it does appear that a large oxygen content has been introduced to high purity silver in the present work and yet has raised the stacking fault energy only to 23.8 ergs/cm^2. In less pure silver it may well be possible to increase the effective value of γ further on adding oxygen as a consequence of a strong solute impedance force arising from oxidized impurity clusters. The non-equilibrium shape of the node in Ag + O_2 in Fig. 2b indicates the presence of a local solute impedance force, arising, despite the high purity of the metal, from an oxidized impurity cluster or possibly from Ag_2O.

A number of different annealing treatments have been performed on the alloy with e/a = 1.10 and high oxygen content. If deformation was introduced at room temperature after furnace cooling from an anneal in air at 500°C, the nodes were ~40% smaller than in the alloy with e/a = 1.10 and low oxygen content. However, if the annealing took place under high vacuum, the nodes were only ~6% smaller than in the low oxygen content alloy, presumably due to the latter treatment eliminating some of the oxygen introduced earlier. The 40% increase in the effective value of γ is very probably due to the presence of an oxidized-solute impedance force which prevents the partials from reaching equilibrium.

Further evidence for oxidized clusters in 99.98% and alloyed copper and pure silver has been obtained by Ashby (1965). In silver the introduction of oxygen increased the temperature dependence of the initial flow stress considerably, and the results could be interpreted as due to particles ~10Å in size, while in copper the effects were rather smaller.

Preliminary results on the temperature dependence of the stacking fault energy in the present alloy series have revealed an irreversible increase in the stacking fault energy above room temperature for specimens with e/a = 1.15 and 1.23. For e/a = 1.15, the mean value of the inscribed radius ($\bar{w}$) of a large sample of nodes decreased by ~25% from its value at room temperature following a 60 minute anneal at 260°C, while for e/a = 1.23 a 60 minute anneal at 400°C led to a 35% decrease in $\bar{w}$. Annealing experiments in a Cu-Al alloy by Christian and Swann (1965) led to similar results to the above and were interpreted in terms of the thermally activated motion of the partials enabling them to overcome the solute impedance force and reach equilibrium.

Because of the presence of a solute impedance force it is premature to attempt to interpret the results of Fig. 2 theoretically by considering only the perturbations caused by the solute additions to the effective atomic potentials which exist in pure silver. However, work in progress is aimed at providing a plot of γ vs. e/a as determined from thoroughly annealed nodes, together with information on the reversible and irreversible temperature dependence of the stacking fault energy throughout the solid solution range. These results, it is hoped, will provide a good basis for comparison with theory.

3.2 Extrinsic Stacking Fault Energy

Figure 3 shows examples of extrinsic-intrinsic node pairs in three alloys. Extrinsic faulting of this type has been reported before by Loretto (1964) and by Ives and Ruff (1966), but only for alloys of very low stacking fault energy. Gallagher (1966b) showed examples of extrinsic-

intrinsic node pairs in Ag-In alloys of higher stacking fault energy. The small network of nodes, all of which are extended, in Fig. 3a is a clear example of extrinsic faulting in an alloy with e/a = 1.008, for which the intrinsic stacking fault energy is 20.7 ergs/cm^2. As in Fig. 1 the difference in the size of the node pairs in Fig. 3a, b and c is very striking.

More quantitative information regarding the magnitude of the extrinsic stacking fault energy in the Ag-In series can readily be obtained from the extrinsic-intrinsic fault pairs reported by Gallagher (1966b), examples of which are shown for three alloys in Fig. 4. Numerous other examples in the alloys with e/a = 1.15 and 1.23 are shown in the above reference, from which it was shown that within close limits the extrinsic (γ_e) and intrinsic (γ) stacking fault energies were equal (γ_e/γ (e/a = 1.15) = 1.09 ± 0.1; γ_e/γ (e/a = 1.23) = 1.03 ± 0.1). The fault pair illustrated in Fig. 4a is the only one observed to date in such a high stacking fault energy material, although several examples have been observed in a Ag-Sn alloy for which $\gamma \sim 15$ ergs/cm^2. The widths of the intrinsic and extrinsic faults are the same to within ~30%, showing that in this alloy too, γ and γ_e are of a similar magnitude. This is such a dilute alloy (0.5wt% In) that it appears very likely that γ is approximately equal to γ_e, not due to the effects of alloying, but more generally including pure metals.

A comparison of the intrinsic stacking fault energy determined from fault pairs and from nodes is made in Fig. 2. The single fault pair in the alloy with e/a = 1.008 gave a value of γ in close agreement with the node value. For e/a = 1.15 and 1.23 the fault pair values are lower

than, but in satisfactory agreement with, those determined from nodes.

Thus, extrinsic faulting is of much more general incidence than has hitherto been supposed. The fact that the extrinsic and intrinsic stacking fault energies are closely the same suggests that close attention whould be paid to the proposal of Weertman (1963) concerning the likelihood of the formation of interstitial producing jogs.

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Table 1

THE INTRINSIC STACKING FAULT ENERGY IN THE Ag-In SERIES, FROM EXTENDED NODE MEASUREMENTS

e/a	$\bar{w}$ (Å)	Δw_1* (Å)	Δw_2* (Å)	number of nodes	$G.10^{11}$ (dynes/cm^2)	ν	γ (ergs/cm^2)	$\Delta\gamma_1$* (ergs/cm^2)	$\Delta\gamma_2$* (ergs/cm^2)
1.00	87	14	3	20	2.64	.42	21.9	3.5	1
1.008	92	12	4	10	2.63	.42	20.7	2.7	1
1.019	92	16	4	16	2.62	.42(5)	20.6	3.5	1
1.037	87	15	4	13	2.61	.43	21.8	3.8	1
1.10	117	18	4	23	2.55	.45	16.1	2.5	0.6
1.15	230	80	16	40	2.50	.46	8.7	2.5	0.6
1.23	412	100	15	40	2.42	.48	5.6	1.3	0.3

*Δw_1, $\Delta\gamma_1$, standard error of a single reading

Δw_2, $\Delta\gamma_2$, standard error of the mean

FIGURE CAPTIONS

Figure 1. Nodes in the Ag-In series, from pure silver to e/a = 1.23.

Figure 2. The intrinsic stacking fault energy (γ) as a function of e/a in the Ag-In series.

Figure 3. Extrinsic-intrinsic node pairs (A),

(a) e/a = 1.008 (γ = 20.7 ergs/cm^2),

(b) e/a = 1.15 (γ = 8.7 ergs/cm^2),

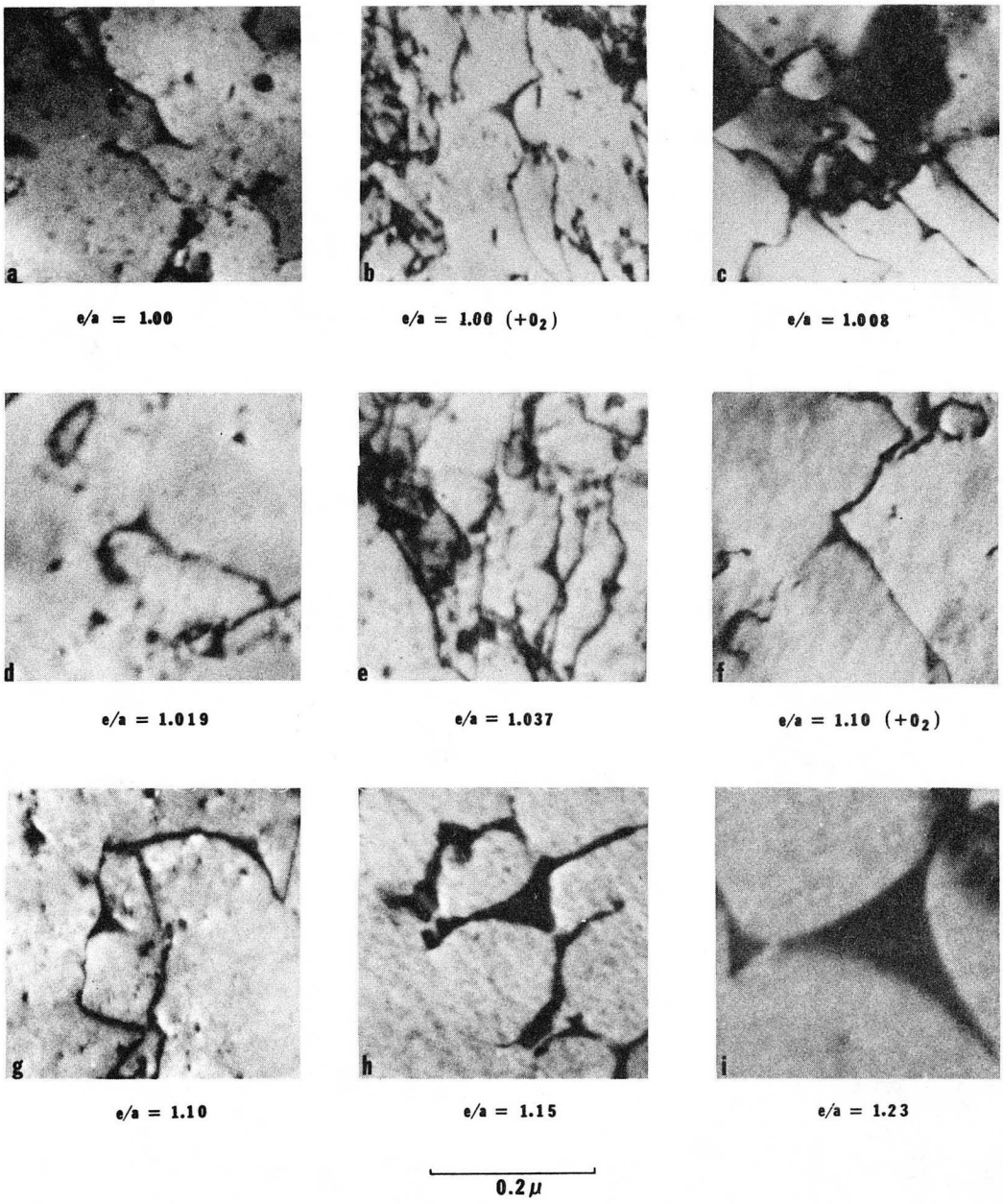
(c) e/a = 1.23 (γ = 5.6 ergs/cm^2).

Figure 4. Extrinsic-intrinsic fault pairs (B),

(a) e/a = 1.008 (γ = 20.7 ergs/cm^2),

(b) e/a = 1.15 (γ = 8.7 ergs/cm^2),

(c) e/a = 1.23 (γ = 5.6 ergs/cm^2).



e/a = 1.00 | e/a = 1.00 ($+O_2$) | e/a = 1.008

e/a = 1.019 | e/a = 1.037 | e/a = 1.10 ($+O_2$)

e/a = 1.10 | e/a = 1.15 | e/a = 1.23

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Fig. 1

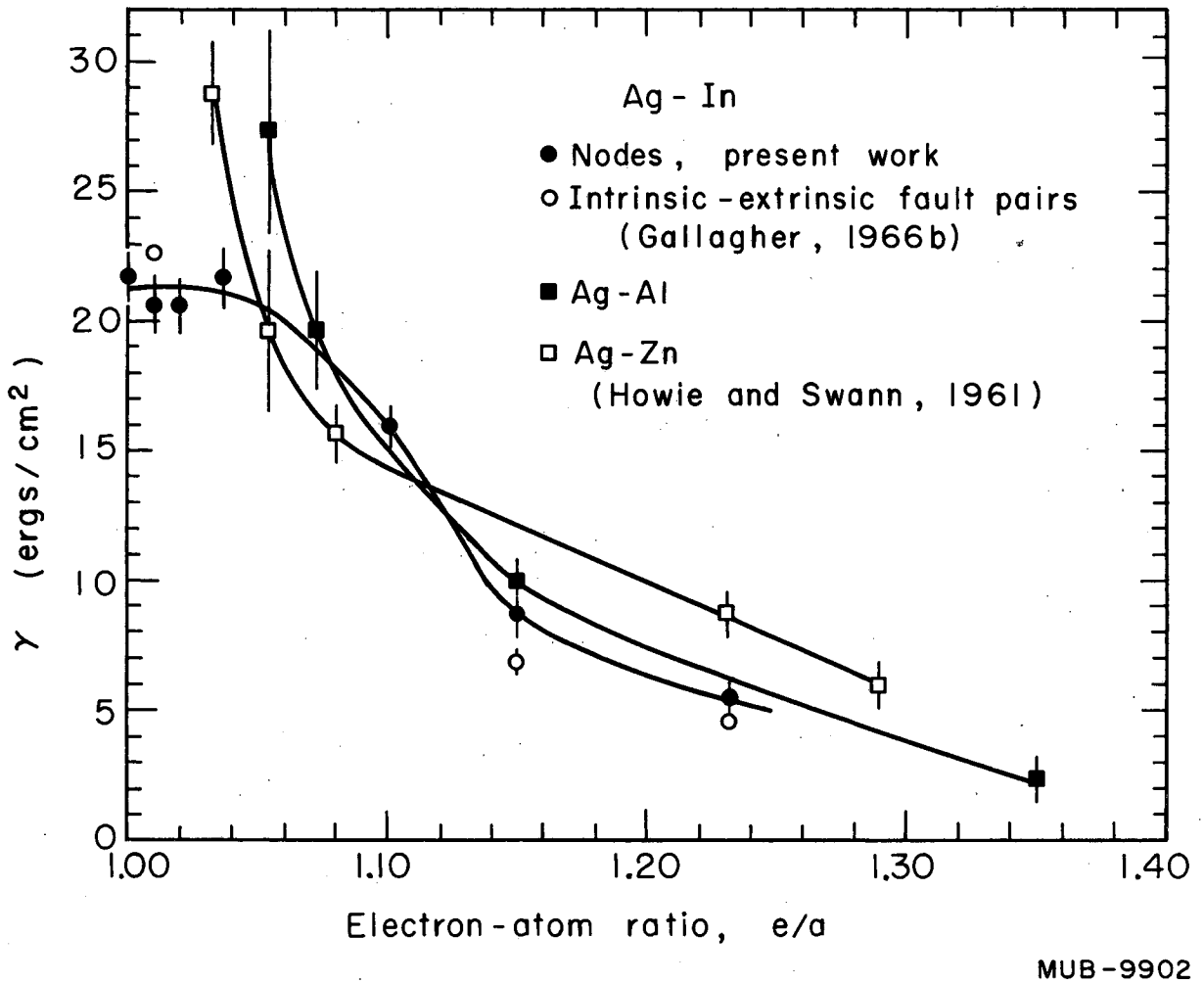
Ag - In
● Nodes, present work
○ Intrinsic - extrinsic fault pairs (Gallagher, 1966b)
■ Ag - Al
□ Ag - Zn (Howie and Swann, 1961)
γ (ergs / cm²)
0
5
10
15
20
25
30
1.00
1.10
1.20
1.30
1.40
Electron - atom ratio, e/a
MUB-9902

Fig. 2

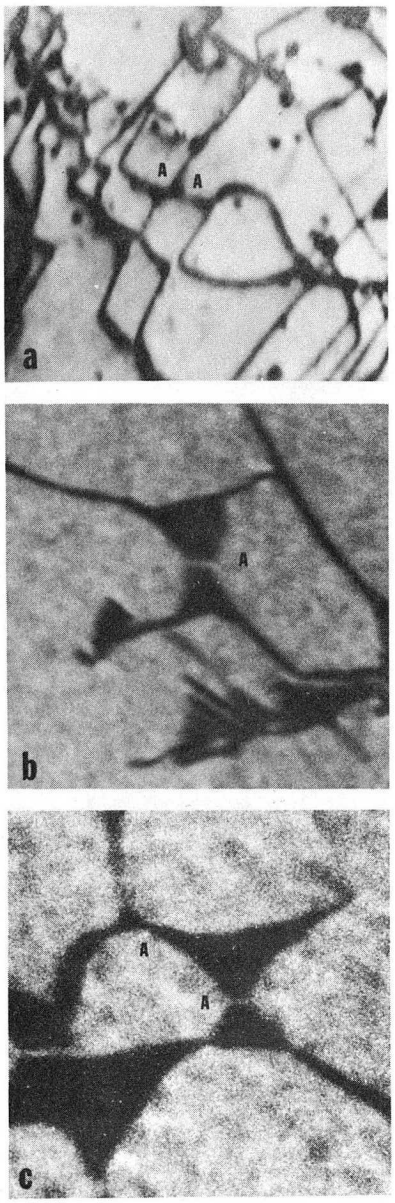

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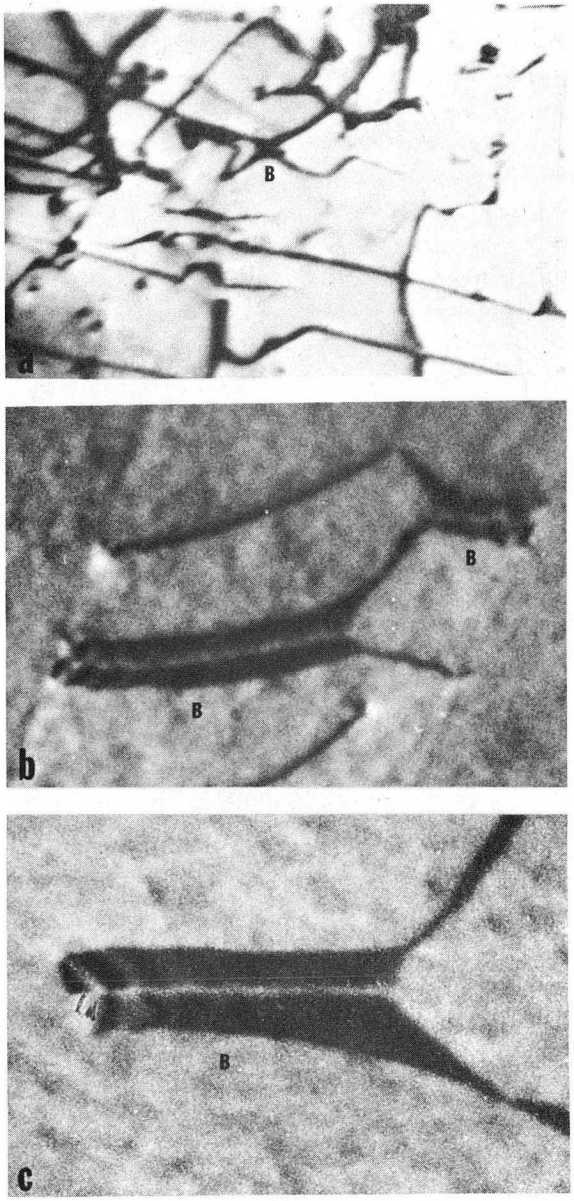
Fig. 3



0.2 μ

ZN-5562

Fig. 4

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