Title
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Publication Date
1975-12-01

Submitted to Physica Status Solidi

LBL-4536 c.1
Preprint c.1

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December 1975

Prepared for the U. S. Energy Research and Development Administration under Contract W-7405-ENG-48

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LBL-4536

THE ORIGIN AND NATURE OF MULTIPLE LOOP CONFIGURATIONS IN QUENCHED ALUMINUM AND ALUMINUM ALLOYS

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ABSTRACT

Multiple loop configurations in quenched aluminum-silicon alloys have been studied using transmission electron microscopy. An analysis of the defect geometries and complex unfaulting behaviour is shown to be consistent with the coplanar loop structure. This is in agreement with a recent electron microscopy contrast analysis of multiple loops in an aluminum-magnesium alloy, but contrary to previous studies on nominally pure aluminum where the layered structure was found. A model is developed in which both the layered and coplanar structures can develop depending on the magnitude of the stresses generated around impurity particles. It is also shown how the coplanar loop structure originates from the operation of a Bardeen-Herring climb source.

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1. Introduction

Dislocation loops in concentric arrangements of varying complexity have been observed frequently in quenched aluminum and aluminum-based alloys [1-3] . In the simplest cases these defects are variants of single faulted Frank loops and take the form of double, triple or quadruple faulted loops generated successively on a {111} plane. The formation of these multiple loops, subsequent unfaulting, and diffraction contrast behavior, has been studied extensively by transmission electron microscopy but a full understanding of their structure and origin has not yet been obtained. Initially, studies performed on double-faulted loops in nominally pure Al [4,5] led to the proposal of two alternative structures which are illustrated in Fig. 1, (in the analysis that follows the loops are designated 1,2 and 3, as shown) the so-called layered structure (1a and b) and the coplanar structure (1c and d). Both were shown to be consistent with the observations but the layered arrangement was considered the most probable from arguments concerning the geometries of the two structures. For example, an explanation for the observed triangular shape of the inner loop, and a logical formation mechanism could be found only in terms of the layered structure.

Subsequent observations of triple and quadruple faulted loop in aluminum [6,7] and other variants of multiple loops in Al-Mg alloys [8] were also best explained by an extension of the layered model, so this became the accepted structure. Recently, however, Eikum and Maher [9] have made a detailed re-examination of the dislocation and fault contrast in the electron microscope both before and during the progressive unfaulting of double and triple loops in an Al-Mg alloy and arrived at interesting conclusions. They were able to show that the Shockley partial nucleated during the unfaulting of the outer (largest) loop interacts with the second loop. In contrast to the earlier work, this behavior can be understood only in terms of the coplanar structure unless a "trapping" mechanism is invoked in which the Shockley and Frank partials interact. Such trapping is, however, considered unlikely since the partials lie on next nearest neighbor planes.

Turning now to the more complex multiple dislocation loop configurations that have been found in certain aluminum-copper and aluminum-magnesium alloys: [10-14] these defects usually consist of concentric perfect loops and are distinguished by almost always having visible inclusions associated with them. They are believed to arise by the operation of dislocation sources either by glide in the manner proposed by Frank and Read [15] or by the analogous climb mechanism suggested by Bardeen and Herring [16] . The geometry and operation of these sources in quenched alloys has been discussed in detail by Smallman and Eikum.[2] In the past a distinction has usually been made between these defects and the multiple faulted loops described earlier. However, Smallman and Westmacott [3] have reviewed all the evidence and pointed out many common features amongst the various complex loop categories. Using the example of aluminum-silicon alloys in which both sources and multiple faulted loops are observed,[17] , they proposed that inclusion particles composed of, or based on, silicon generate differential strains in the matrix during quenching and thus constitute preferential sinks for vacancies. The magnitude of the stresses developed at the particle/matrix interface will depend on the particle size and misfit, and if sufficiently large could lead to the nucleation of dislocation segments which subsequently act as sources.

In the present paper these concepts are developed further and additional evidence for the coplanar structure is given. Subsequently a simple model is proposed which is capable of explaining in detail most of the available facts on the structure of all the multiple loop configurations.

2. Experimental Details

Previous work has shown that in Al-Si alloys the incidence of multiple loops is much greater than in other Al-based alloys. [17] In order to investigate these effects further a series of alloys containing 0.05, 0.25, 0.5, 0.75 and 1% Si was prepared from a master Al-1.3% Si alloy. After adequate homogenization treatments, foil samples were prepared and quenched from air using a conventional vertical tube furnace arrangement. The large loops required for this study were formed by quenching from 550-600°C and aging in silicone oil heated to ~100°C. To prevent unfaulting of the loops during specimen preparation, all samples for electron microscopy were prepared by a chemical polishing technique which minimizes handling. [17] The observations were made on a Siemens I operated at 100kV.

3. Results

3.1 General Observations

In all the alloys studied high concentrations of perfect and imperfect prismatic dislocation loops of varying complexity were observed (see Fig. 2). The loop distributions were usually uniform and consisted of mixtures of single loops and concentric double and triple loops. The incidence of double and triple loops, and the ratio of perfect to imperfect loop concentrations, both increased with silicon concentration. The changes in loop character have been studied previously [17] and explained by a model in which progressive loop unfaulting occurs as increases in the solute concentration produces changes in stacking fault energy and internal stress distribution.

In alloys containing more than about 0.75% Si many examples of multiple loops were observed in a configuration which bear a strong resemblance to that expected from a classical Bardeen-Herring climb source (for example at A in Fig. 3 where the "kidney-shape" inner loop is clearly seen). Furthermore, in many cases it is apparent that the innermost dislocations are associated with a particle which occasionally is large enough to be resolved in the electron microscope (see example A in Fig. 3).

3.2 Loop Unfaulting Behavior

In specimens taken from material containing more than 0.25% Si many of the double and triple loops were in various stages of unfaulting. The observed contrast and apparent unfaulting behavior of these defects was not inconsistent with previous analyses of multiple loops [6,7].

In some instances, however, unusual dislocation arrangements were observed, and two examples together with schematic diagrams which clarify their structure are given in Fig. 4. These examples show an intermediate stage in the unfaulting of double, 4(a), (b), and triple, 4(c), (d), loops respectively. Both micrographs show partially unfaulted multiple loops in which (i) loop 1 has unfaulted but not loop 2, (ii) the unfaulting partial dislocation has interacted with both loop 1 and loop 2, and (iii) loops 1 and 2 are joined by additional dislocation segments. In Fig. 4(a) and (b) this extra dislocation bisects the loops, while in the triple loop example {Fig. 4 (c), (d)} it asymmetrically divides the loops. It should also be noted that in Fig. 4(c) loop 3 is unaffected by the unfaulting of loop 1. Consideration of this arrangement suggests that unfaulting of the region between loop 1 and 2 has occurred by the nucleation of two different Shockley partials {S_1 and S_2 in Fig. 4(b)}. Similar "double Shockley" nucleation in the unfaulting of single Frank loops in Al-Mg alloys has recently been reported [18]. On one occasion the unfaulting of the outer region of a double faulted loop was observed directly in the microscope (see Fig. 5) but the nucleation and reaction of the Shockley partial dislocations could not be followed. In the example loop A has transformed during the interval between the taking of the two micrographs, about 2 minutes, and the changes in loop configuration are seen to be exactly the same as those illustrated in Fig. 4.

In analysing these structures the loop plane is defined as (111), (i.e. the loop Burgers vectors are initially $a/3[111]$), the possible unfauling Shockley partials are therefore $a/6[11\bar{2}]$, $a/6[\bar{2}11]$ and $a/6[1\bar{2}1]$; the dislocation line sense is taken as shown in Figs. 4(b) and (d). Each pair of Shockley partials produces the required configuration, but with a given $g = [111]$

diffraction vector operating only two of the three pairs satisfy the observed contrast behavior. An example of the complete analysis which is consistent with the contrast behavior shown in Fig. 4(a) in which the electron beam direction z=321, and $g=\pm[1\bar{1}\bar{1}]$ is given in the following Table:

Dislocation Segment	Dislocation Reactions and Products	g.b	Contrast Theory	Exp.
A'A	$a/3[111]+a/6[11\bar{2}] \rightarrow a/2[110]$	0	out	out
B'B	$a/3[111]-a/6[11\bar{2}] \rightarrow a/6[114]$	±2/3	in	in
AA'	$a/3[111]+a/6[\bar{2}11] \rightarrow a/2[011]$	± 1	in	in
BB'	$a/3[111]-a/6[\bar{2}11] \rightarrow a/6[411]$	±1/3	out	out
AB B'A'	$a/6[11\bar{2}]-a/6[\bar{2}11] \rightarrow a/2[10\bar{1}]$	± 1	in	in

The dislocation reactions at the nodes A,A',B,B', must also satisfy the condition $b_1+b_2+b_3=0$. for example at node A with the line senses as shown

$$a/2[110] - a/2[011] - a/2[10\bar{1}] = 0$$

The same result is found for the nodes at B,B' and A'.

Exactly the same analysis is consistent with the observed behavior of the double loop of Fig. 5b, and loop of Fig. 4(c). In this case, z=[101], $g=\pm[1\bar{1}\bar{1}]$, and the micrograph was taken in dark field under g(-g) diffraction conditions.

4. Discussion

In the present study of multiple loops in quenched Al-Si several examples of double and triple loop unfaulting by the simultaneous nucleation of two Shockley partials have been observed. Analysis of the results show that unfaulting of the intrinsic region between loops 1 and 2 leaves the extrinsic faulted inner region unaffected, i.e., the dislocation reactions occur at loop 2. This behavior is consistent with the coplanar loop structure of Fig. 1(c) and (d) but cannot be readily explained on the layered model. The same conclusion was reached recently by Eikum and Maher after performing detailed contrast analysis on double and triple loops in an Al-3% Mg alloy.

The second notable feature of the present work is the frequent occurrence in concentrated Al-Si alloys of multiple loops in which the centre regions are frozen in classical Bardeen-Herring climb source configurations. In the next section it will be shown how the operation of such a source can lead to the development of the coplanar structure.

In contrast to this conclusion that multiple loops have the coplanar structure, all previous workers have found compelling evidence that supported the layered structure model. This evidence includes, (1) the observation of triangular-shaped inner loops which is expected only with the layer model, (2) observed differences in stacking fault and loop contrast before and after unfaulting, and (3) the occurrence of multi-component loops consisting of triangular loops on either side of the original hexagonal loop which coalesce during annealing [8]. It is noteworthy, however, that the layered structures have been found in nominally pure aluminum and dilute alloys, whereas the coplanar structures are found in alloys in which the solute element is supersaturated at room temperature. This is taken into account in the next section where a model is developed in which both the layered and coplanar structures can form depending on the alloy composition.

4.1 Model for Multiple Loop Formation

It is now generally accepted that multiple loops are heterogeneously nucleated on particles or inclusions but substantive evidence on the nature of the inclusion is scant. Gulden and Nix [11], in a study of glide loop sources in Al-Cu, were able to identify large particles in the center of each source as elemental silicon and traced their origin to the quartz tubes used to prepare the alloys. Furthermore, in the present work the incidence of multiple loops in the Al-Si alloys was extremely high. Therefore, in the absence of evidence to the contrary, it will be assumed that multiple loops form in association with Si or SiO_2 particles accidentally or deliberately introduced into Al. However, in different alloy systems other inclusions can play a similar role. On quenching such a material the large differential expansion between Si and Al (3×10^{-6} c.f. 25×10^{-6}/°C) produces large compressive stresses in the matrix surrounding the particle. The original treatment of the stress distribution around particles was given by Eshelby [19], and many subsequent workers have considered how relief of the stress might be accomplished [e.g. 20 to 24]. According to Ashby [25], dislocation nucleation at incoherent interfaces is a relatively easy process and requires stresses of about G/100 (where G is the shear modulus). In the present context, only the treatment of Gulden and Nix, who considered the case of glide loop nucleation at the particle/matrix interface, is applicable. By equating the condition for the expansion of a glide loop with the maximum shear stress developed at the particle interface these authors were able to show that a minimum particle size exists below which a shear loop cannot nucleate and grow. For the conditions obtaining in a quenching experiment a value for the minimum particle radius, $r_{o(min)} = 30Å$

was found; however, when a strain rather than a stress criterion was used r_o increased to ~ 200A. It was also pointed out that a complete glide loop can form only if an external shear stress is superimposed on the differential compressive stress. In the present experiments very slow oil quenching which minimise quenching stresses was used and under these circumstances shear loops may be unable to form. Stress relief may then occur by the nucleation of a segment of sessile dislocation which can subsequently operate as a climb source. To examine the feasibility of this we follow the Gulden and Nix treatment to estimate the normal stresses at the particle interface. Assuming a spherical incoherent particle, the stress at the interface, σ_{ii} is given by

$$\sigma_{ii} = 4\alpha\Delta TG$$

where α is the difference between the expansion coefficient of the particle and matrix, and ΔT the change in temperature during the quenching. Taking $\alpha \simeq -20\times10^{6}/°C$ and $\Delta T \simeq 500°C$, $\sigma_{ii} \simeq -G/25$. This is a very high stress indeed, and according to an estimate by Kelly [21] very close to the theoretical strength of a crystal. However, as noted above, if in simple strain criterion is used by setting $\alpha\Delta Tr_o = b$[11] a particle size dependence is found. According to this relation, for the same experimental conditions, the minumum particle size for the nucleation of a complete loop is ~200A, as before.

If this approach is valid the prediction of a particle size dependence forms the basis for explaining the occurrence of both types of multiple defect structure around the particle. Thus when particles are present of a size less than the critical radius, dislocations will not be nucleated but the regions of the matrix under compressive stress will still constitute a preferential sink for excess vacancies formed during the quench. After the first loop has nucleated and grown it will be indistinguishable from a Frank loop nucleated in the stress free-matrix. However, if the stress persists as the first loop climbs out of the stressed region successive loops will nucleate and grow concentrically with the first one. These loops will not be constrained to lie on particular planes and will therefore adopt the lowest energy structure which is believed to correspond to that of the layered array of Fig. 1(a) and (b).

Consider now the condition where particles are present with $r>r_{crit}$ and a segment of sessile dislocation is nucleated on a {111} plane. The Burgers vector is a a/3[111], and if the segment extends around the particle periphery, part of it will be of edge character while the ends will have a screw component. It therefore satisfies the geometrical conditions to act as a climb source in the manner proposed by Bardeen and Herring[16]. Under the high chemical stress of the excess vacancies produced by the quench, several complete loops will be generated before the line tension of the source dislocation exceeds the diminished chemical stress and climb terminates. We now show that operation of the Bardeen-Herring source leads to the coplanar structure of Fig. 1(c) and (d).

4.2 Geometric Structure of Climb Source

The sequence of events during the operation of a source is illustrated schematically in Fig. 6; the cross and dashed line represent the end-on view of the source which is operating on the (111) planes shown in cross-section. After operating once {Fig. 6(a)} a normal Frank loop has been generated and the source dislocation has climbed up to the next nearest plane. When the second loop is generated its growth leads to BB stacking which constitutes a high energy fault {Fig. 6(b)}. However, Shockley partial dislocations are readily nucleated in aluminum and the high energy fault is reduced to an extrinsic fault by the nucleation of a Shockley partial dipole. The dipole, which propagates above and below the B plane, changes the stacking of this layer only and does not change the loop Burgers vector. Thus the structure is as shown in Fig. 6(c) and consists of a coplanar annulus of intrinsic fault surrounding an extrinsic loop. This is precisely the coplanar structure of Fig. 1(c). A similar process occurs during the formation of a third loop {Fig. 6(d) and (e)} where nucleation of the Shockley dipole now restores the inner loop region to perfect stacking. If the vacancy supersaturation is still sufficient to produce loops a further set of three will nucleate in the next nearest (111) plane as indicated in Fig. 6(f).

To summarize, it is proposed that [1] all multiple loop configurations are heterogeneously nucleated at inclusions in the matrix, and (2) the structure of the multiple loops which form on quenching depends on the size of the inclusion. When sub-critical size particles are present the compressive stresses are relieved by the condensation of successive vacancy loops in the layered structure. On the other hand, when the particles exceed the critical size, sessile dislocation segments nucleate and act as Bardeen-Herring sources. This leads to the development of the coplanar structure. If very large inclusions are present glide or climb sources may be generated on one or more crystallographic systems. This would lead to the development of the complex arrangements observed in certain Al-Mg and Al-Cu alloys that have been described in detail previously[2].

4.3 Unfaulting of Loops in Coplanar Configuration

The unfaulting of loops condensed in the layered structure has been described in detail by Edington and West [6,7].

In the case of the coplanar structure the unfaulting reactions are completely analogous to those of the layered configuration, the only differences being in the number and sequence of Shockley partials propagated. Consider first the double fault structure of Fig. 6 (c); an unfaulting Shockley partial propagated between loops 1 and 2 must react with dislocation loop 2 as well as 1 according to a reaction of the type

$$a/3[111] + a/6[11\bar{2}] \rightarrow a/2[110] \qquad \text{at loop 1}$$

and

$$a/3[111] - a/6[11\bar{2}] \rightarrow a/6[114] \qquad \text{at loop 2}$$

Since the inner extrinsically faulted region is unaffected by this reaction no change in the inner fault contrast will be observed. This absence of a fringe shift was a notable feature of the Eikum and Maher observations, and is in marked contrast to earlier work on nominally pure Al in which clear fringe displacements were observed after unfaulting [6,26]. Subsequent unfaulting of loop 2 occurs by the propagation of two Shockley partials above and below the fault by a reaction of the type,

$$a/6[114] + a/6[11\bar{2}] - a/6[\bar{1}\bar{1}2] \rightarrow a/2[110]$$

Thus the final configuration consists of two concentric perfect prismatic loops as in the case of the layered structure.

A similar reaction will produce unfaulting of a triple loop, but in this case the dislocations combine at loop 3 according to a reaction of the type

$$a/3[111] - a/6[11\bar{2}] + a/6[\bar{1}\bar{1}2] \rightarrow a[001]$$

The complete set of nine possible combinations of Shockley partials for the unfaulting of triple loops has been given by Edington and West[6].

The significant difference in the unfaulting behavior is the interaction of the partial at loop 2 in the coplanar case. All the evidence from the double-Shockley partial unfaulting behavior is consistent with the defects having this structure. An alternative explanation, given by Eikum and Maher[9], that the loops are in fact arranged in the layered structure, and the apparent dislocation reactions at loop 2 result from trapping of the partial as it crosses the Frank dislocation, is not corroborated by the present work. Thus, the fact that in Fig. 4 the unfaulting partials have combined over part of their length (AA' and BB') show that the trapping would have to be very strong indeed to balance the stacking fault and line tension forces acting on the remaining segments (B'B and BB').

4.4 Miscellaneous Observations

We now consider the adequacy of these concepts for explaining other instances in which multiple loops have been observed. Wolfenden[27] has shown that multiple loops are formed in Al irradiated in a high voltage electron microscope. Under these conditions high concentrations of point defects (Frenkel pairs) are produced by the electron beam and these may condense as vacancy or interstitial loops. Wolfenden noted that a high incidence of multiple loops formed in Al which was pre-injected with helium to a concentration 8 atomic ppm prior to irradiation. Since Helium is insoluble in Al it is highly probable that some clustering of the Helium will occur. The large strains around such a cluster will exert a compressive stress in the matrix which can be relieved by vacancy loop condensation. Unfortunately, no determination of the nature of the multiple loops was made.

Urban[28] has also found large multiple-faulted interstitial loops in electron-irradiated nickel, and in his published micrograph (Fig.10 of Reference 28) the characteristic source geometry is clearly visible. Significantly, these complex defects were found only in nickel which had been oxidised prior to irradiation, which is again consistent with heterogeneous nucleation on oxide particles. Similarly, double-faulted loops have been observed in proton-irradiated nickel foils containing significant amounts of oxygen[29].

5. CONCLUSIONS

Unusual unfaulting behavior of double and triple dislocation loops in quenched Al-Si alloys is explained by the simultaneous nucleation of two different Shockley partials.

The results are compared with the two models proposed for the multiple loop structure and, in agreement with another recent study, but contrary to earlier conclusions, found to be consistent with the coplanar configuration. The formation of the coplanar structure is shown to be a logical consequence of the operation of a classical Bardeen-Herring climb source.

A model is proposed in which all multiple loops are heterogeneously nucleated at inclusions, and the occurrence of the coplanar and layered structures depend on whether or not a sessile dislocation is nucleated.

ACKNOWLEDGEMENTS

I wish to thank Dr. H. W. Hunter and Mr. F. C. Essig for encouragement and support through N.W.C.I.R. funding, and Professor G. Thomas for his hospitality and support during my tenure as Visiting Scientist at the Lawrence Berkeley Laboratory, University of California. Thanks are also due to Drs. D. R. Clarke, H. P. Karnthaler and G. Thomas for suggesting improvements to the manuscript, and Mr. Roger Peck for help with the experiments.

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FIGURE CAPTIONS

Fig. 1. Schematic diagrams showing multiple loops condensed in the layered structure (a), (b) and the coplanar structure (c), (d) for double loops (b),(d) and triple loops (a), (c). The loops are designated 1,2,3 in order of decreasing size.

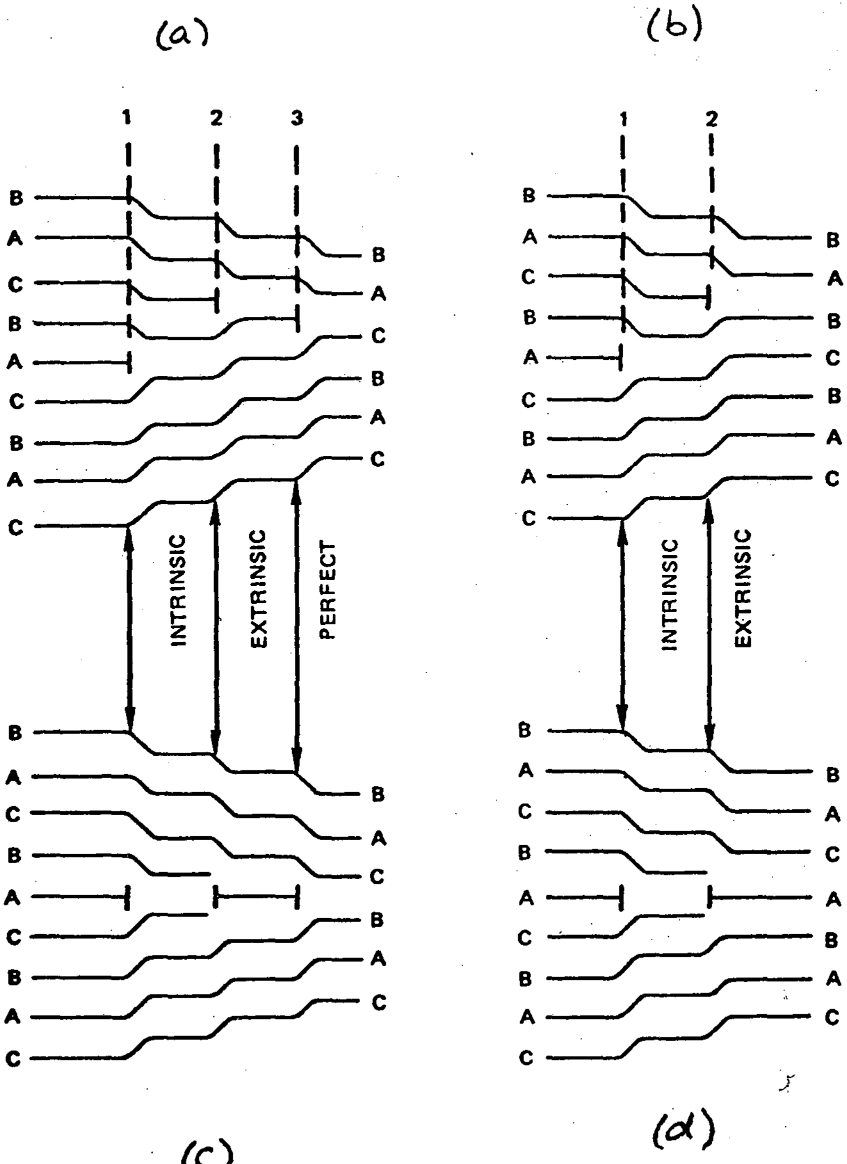
Fig. 2. Series of micrographs illustrating the changes in dislocation loop structure in Al-Si alloys with increasing Si concentration, (a) = 0.05%, (b) = 0.25%; (c) = 0.5%; (d) = 1.0%Si. Note the increase in the concentration of multiple loops with increasing Si content.

Fig. 3. Micrograph illustrating the unusual geometry of certain multiple loops in an Al-0.75% Si alloy. Note the "kidney-shaped" inner loop A,B, and the inclusion in the center of the loops at A.

Fig. 4. Electron micrographs and schematic diagrams showing the dislocation configurations of a partially unfaulted double loop in Al-0.25%Si (a), (b); and triple loop in Al-0.5%Si (c), (d).

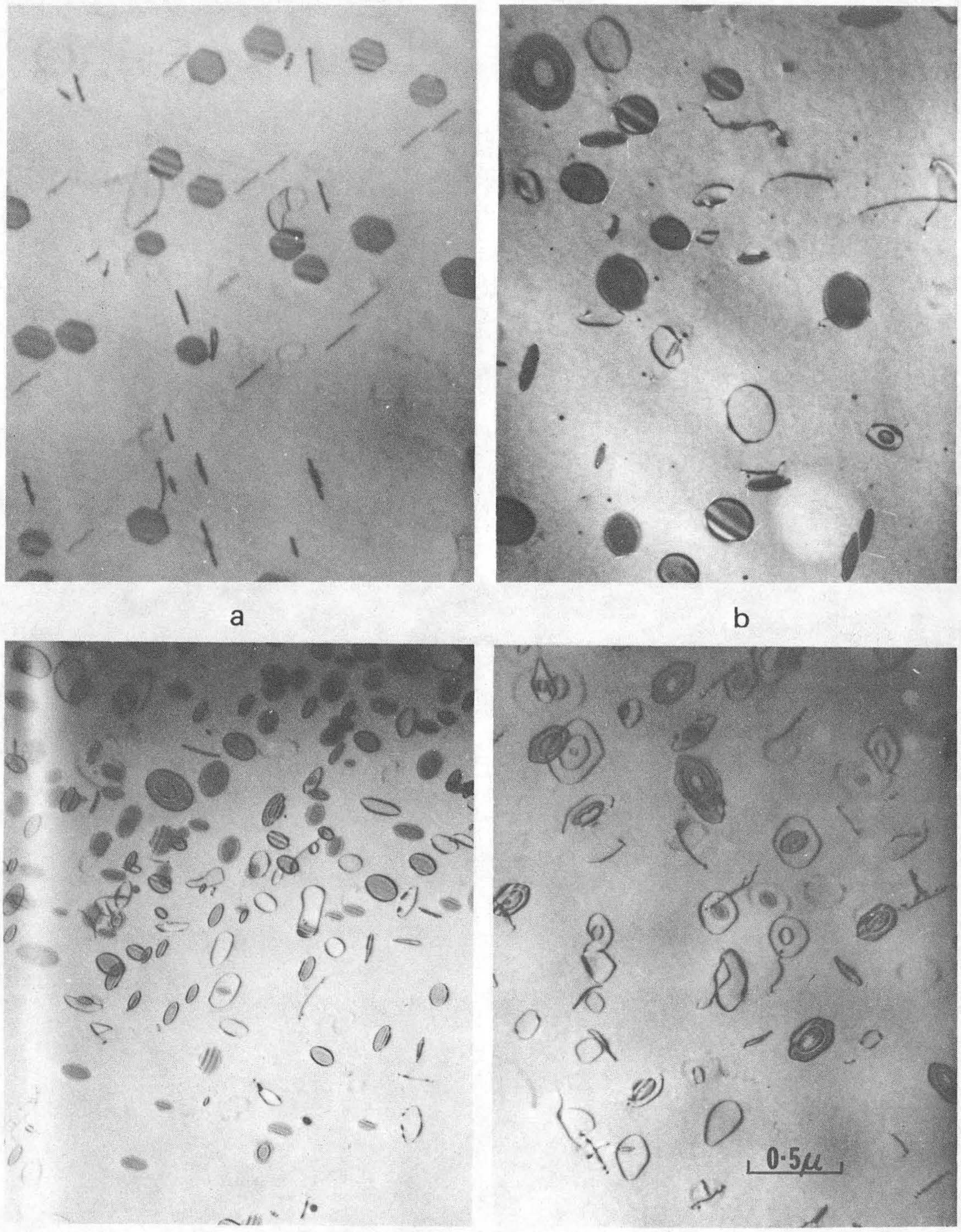
Fig. 5. Sequence of micrographs showing unfaulting of the outer region of a double loop in Al-0.25%Si by the nucleation of two different Shockley partial dislocations. Micrograph (b) was taken two minutes after (a).

Fig. 6. Diagrams illustrating the development of the coplanar loop structure by the operation of a Bardeen-Herring climb source. See text for detailed description.



XBL 764-1131

Fig. 1



a b

c d

Fig. 2

XBB 763-2981

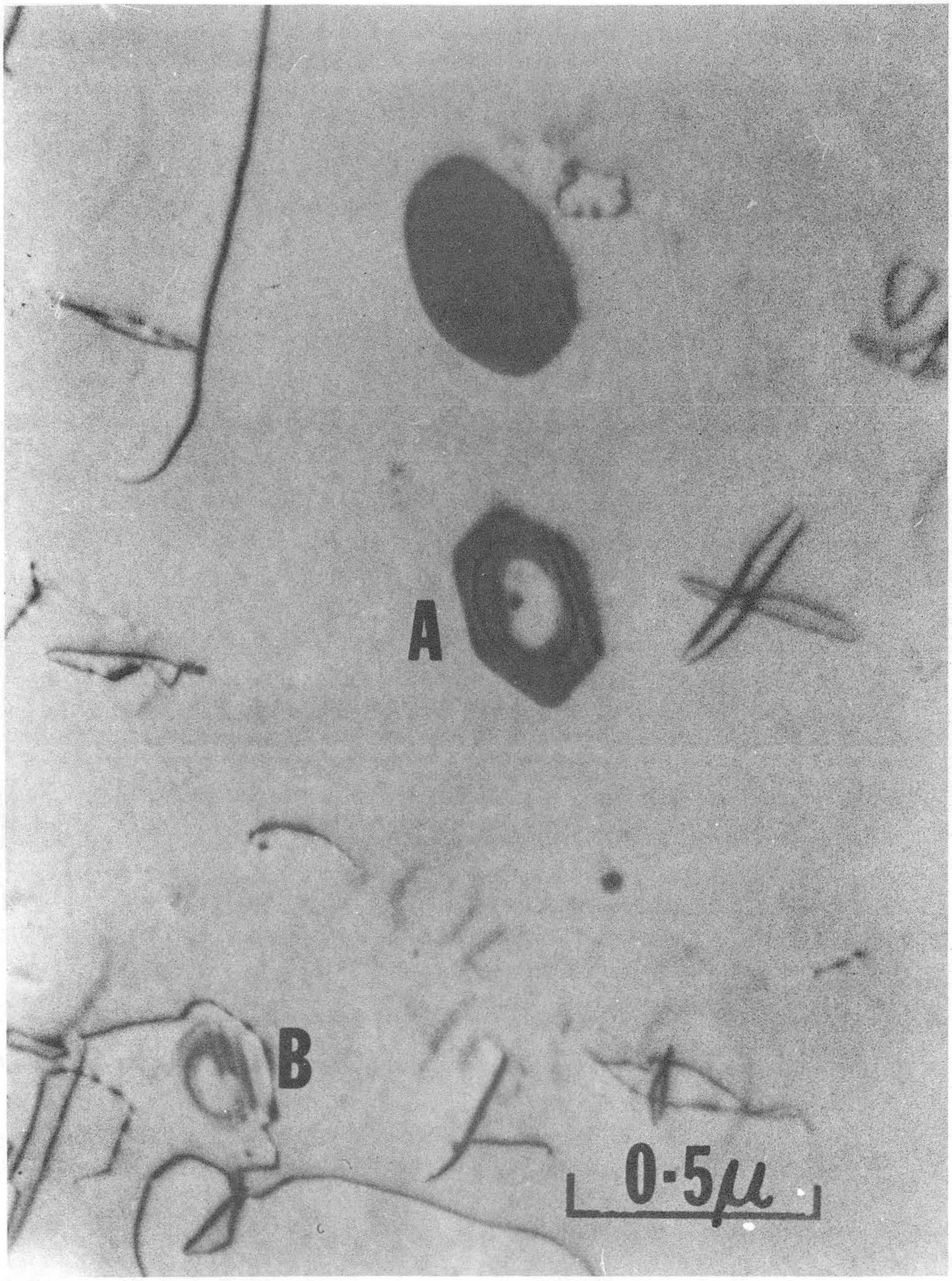


Fig. 3

XBB 763-2983

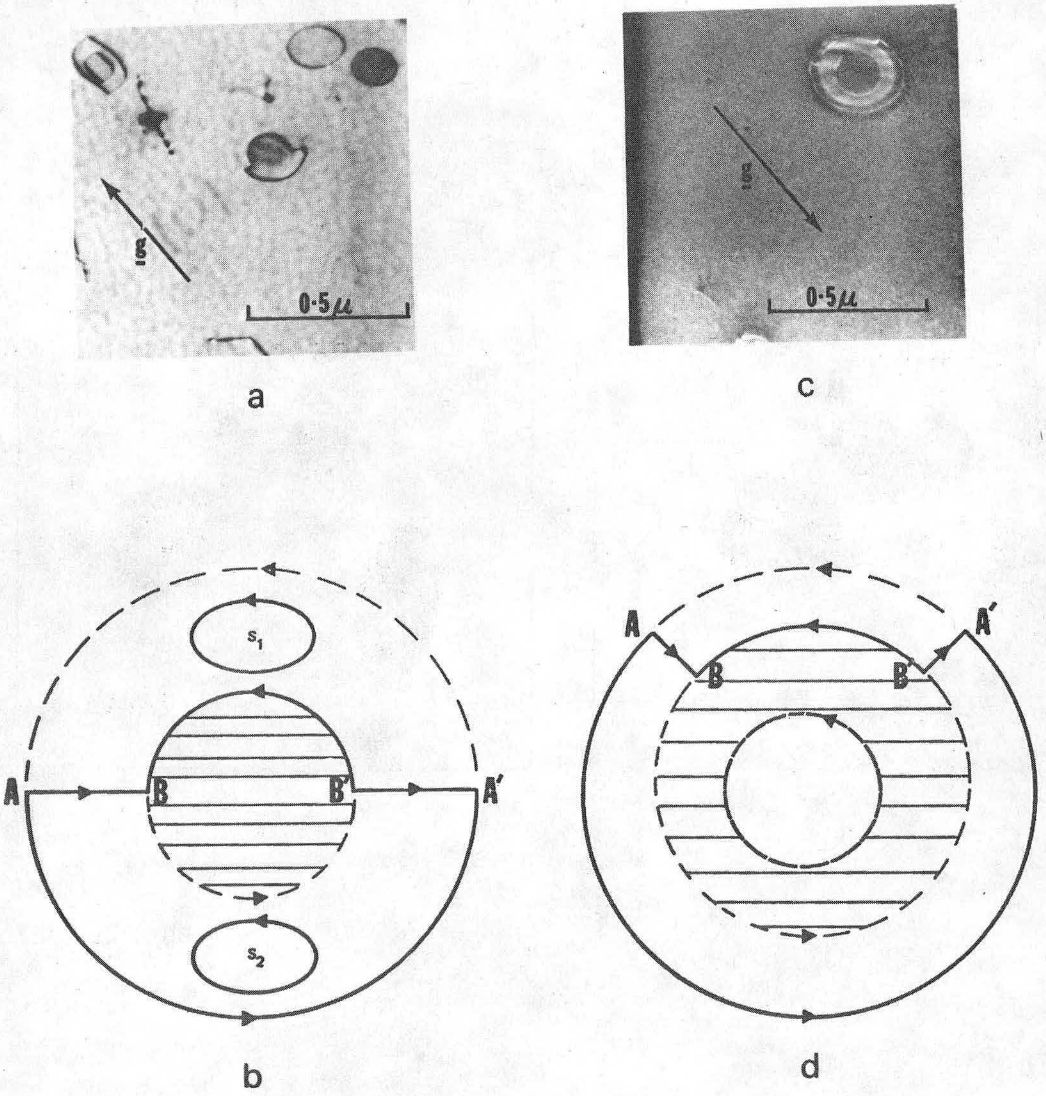


Fig. 4

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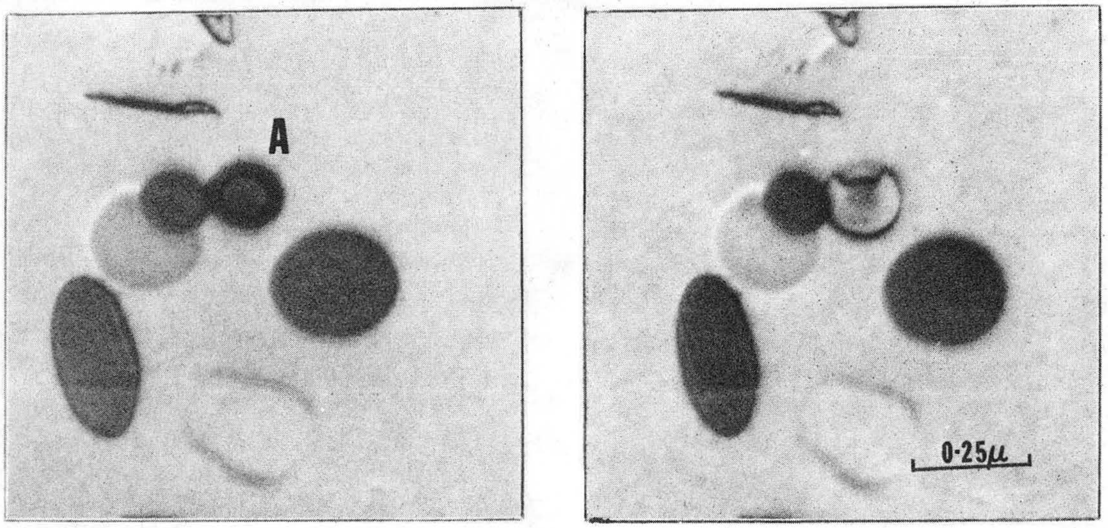

a b

Fig. 5

XBB 763-2982

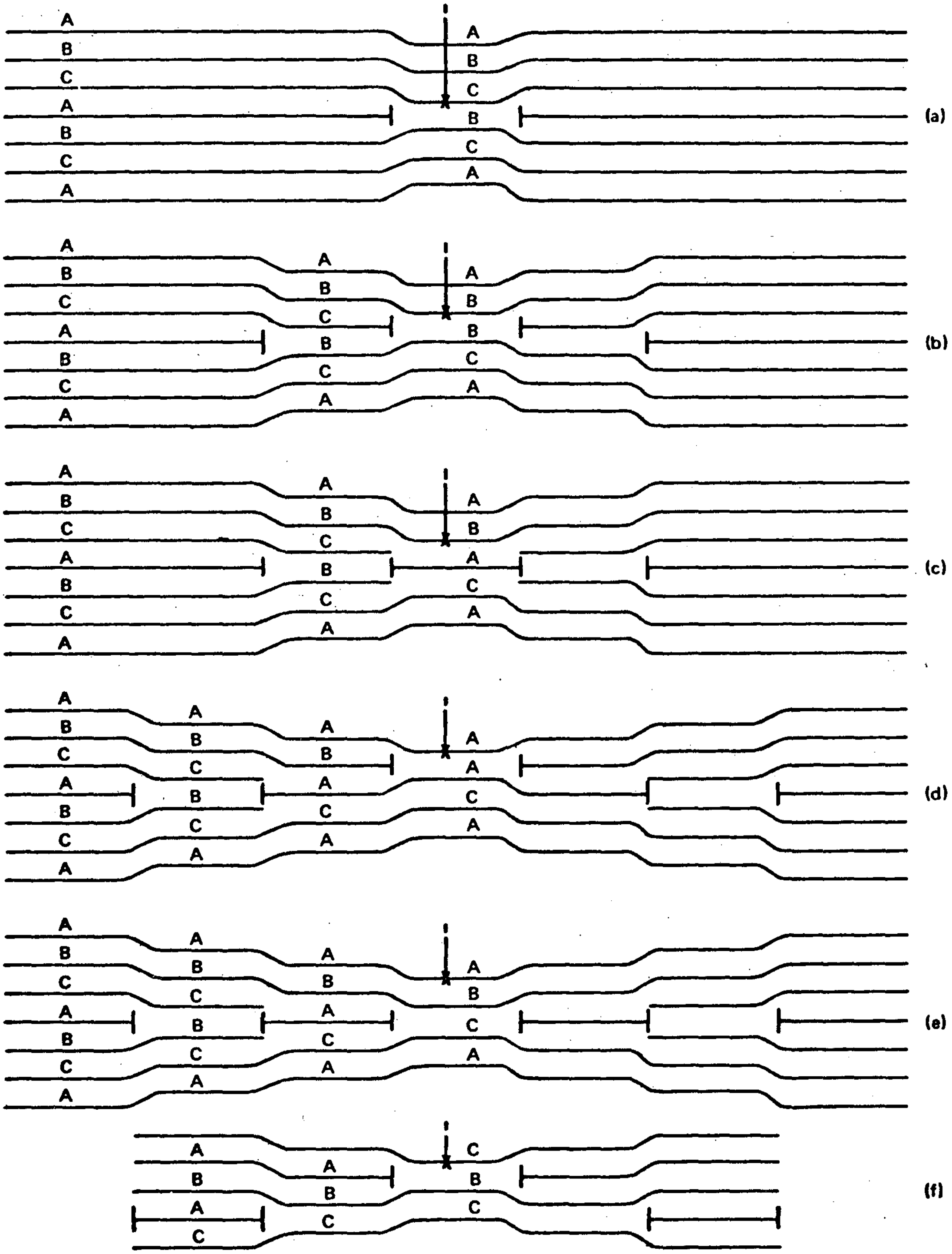

Fig. 6

XBL 764-1132

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