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THE MEASUREMENT OF THE STACKING FAULT ENERGY IN COPPER, NICKEL AND COPPER-NICKEL ALLOYS

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ABSTRACT

There is evidence that the hydrogen solubility and the hydrogen embrittlement of high strength, high performance face centered cubic alloys can be related to the stacking fault energy of the alloys. The stacking fault energy is inversely related to the distance between the two partial dislocations which are formed by the dissociation of a perfect dislocation. The two partial dislocations define a stacking fault in the crystal which offers a region for hydrogen segregation.

To examine this hypothesis, the distance between the partial dislocations will be measured using weak beam, dark field transmission electron microscopy. From these data, the stacking fault energy will be calculated. Initially pure copper, pure nickel and copper-nickel single crystals will be used to determine the stacking fault energy. With the development of the technique, the research will be extended to include high performance alloys for which there are data on hydrogen embrittlement.
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INTRODUCTION

In face centered cubic metals, the perfect dislocation has a burger's vector, \( \mathbf{b}_1 = \frac{3}{2}[110] \). It has been observed that such a dislocation dissociates into two partial dislocations, \( \mathbf{b}_1 \rightarrow \mathbf{b}_2 + \mathbf{b}_3 \) where \( \mathbf{b}_2 = \frac{3}{6}[211] \) and \( \mathbf{b}_3 = \frac{3}{6}[121] \). Since the energy associated with a dislocation is proportional to the square of burger's vector, one can readily show that \( \mathbf{b}_2 > \mathbf{b}_3 \), hence the dissociation of a perfect dislocation is the preferred state.

Dislocation in the face centered cubic lattice lie on the \( \{111\} \) planes. If one makes a traverse through the face centered cubic lattice in a direction normal to a \( (111) \) plane, i.e. \( [111] \) direction, one observes that the atoms are closed packed forming a triangle array in the \( (111) \) plane but the sequence from adjacent planes is different. In fact, there are three possible positions for the planes of atoms which can be labelled a, b, and c. The normal stacking sequence can be given as \( \ldots a \ b \ c \ a \ b \ c \ldots \). If the stacking sequence does not follow this plan, then a stacking fault is present, which results in an increase of the energy of the crystal.

When a perfect dislocation is dissociated into two partial dislocations, the stacking sequence in the region between the two partial dislocations is disrupted hence there will be a stacking fault between the two partial dislocations. The energy of stacking fault, \( \gamma \), is inversely proportional to the distance, \( d \), between the partial dislocations.

There are several properties of metals that depend upon the stacking fault energy. The stacking fault provides a location for possible hydrogen accumulation which may lead to the embrittlement of high strength, high performance alloys. Textures generated by cold working are strongly dependent upon the stacking fault energy of a metal. The ability to undergo cross slip is dependent upon the stacking fault energy. Cross slip is the movement of a dislocation in a face centered cubic metal from one slip plane to another. This process is strongly dependent upon the distance between the two partial dislocations. If the stacking fault energy is high, the stacking fault is narrow and cross slip is easy. Aluminum is a good example. Conversely, low stack fault energy metals do not cross slip easily because the distance between the partial dislocations is large. Austenitic stainless steel is a good example of an alloy with a low stacking fault energy. This alloy works hardens very rapidly.
Objectives

The initial goal of the project is to determine the stacking fault energy for pure copper, pure nickel and copper-nickel alloys. As the techniques are developed, the effort will be extended to include various high strength, high performance alloys. The resulting data will be correlated with existing information of hydrogen solubility and hydrogen embrittlement.

a. Stacking Fault Energies

The initial experimental methods for determining the stacking fault energy have been indirect and somewhat limited in their precision. Low angle x-ray diffraction of filed, severely deformed metal samples can be used to calculate stacking fault probabilities. A second method relies upon the shape of the shear stress-shear strain curve for a single crystal. State I of the deformation represents single slip, i.e. the parallel movement under applied stress of dislocations on parallel slip planes with only one slip system operating. Stage II, the work hardening stage, involves multiple slip in which dislocation on intersecting planes interact giving rise to tangles. This stage continues as long as dislocation multiplication continues and dislocation movement is not blocked. Stage III occurs when the applied stress is high enough to force dislocations to cross-slip. The value of the shear stress at the beginning of Stage III, \( \tau_{III} \), can be used to calculate the stacking fault energy.

With the advent of the 100kV electron microscope, more direct methods of stacking fault determination became available. For metals with high stacking fault energy such as aluminum and the hexagonal close packed metals, zinc and magnesium, quenching from near the melting point will produce vacancy loops. The annealing kinetics of these loops may be used to calculate the stacking fault energy. This technique has been extensively applied by Professor Ray Smallman of the University of Birmingham, England. This method can not be used on metals with low stacking fault energy for the loops that are formed by quenching are irregular in shape and they are not planar so the annealing kinetics for the vacancy loops are difficult to interpret. As an alternative for low stacking fault energy metals, dislocation node size and geometry can be used to obtain the necessary data. A dislocation node is the junction of three dislocations. Unfortunately, there is no metal on which both experimental techniques can be used to obtain the value of the stacking fault by both techniques which will allow a cross check between the techniques.

b. Weak Beam, Dark Field Electron Microscopy

The insertion of a crystal specimen into the electron beam of a transmission electron microscope results in a diffraction pattern. Since this gives very little information, the objective aperture must be inserted which normally is used to block all the diffracting beams, leaving only the transmitted beam resulting in bright field electron microscopy.
There are instances in which one would like to examine one of the diffracted beams. This is done by inserting the Selected Area Diffraction (SAD) aperture to define an area of interest. The objective aperture is removed and the necessary lens currents are adjusted to give a diffraction pattern. By tilting, one can achieve a very bright diffracted beam in contrast to all the other diffracted beams (the two beam condition). This the case when the \( K_o \) vector for the transmitted beam and \( K_{hk1} \) vector for the diffracted beam coincide with the Ewald Sphere. In this case, the value of \( \bar{s} \), the deviation from the reflecting sphere, is zero or nearly zero. By containing the diffracted beam with the objective aperture and removing the SAD aperture one now has a strong beam, dark field image which can be magnified and examined. The image appears to be the same as for bright field illumination except there is an intensity reversal.

Cockayne, Ray & Whelan (1) developed a technique for studying the dislocation strain fields using weak dark field. They use diffraction vector \( \bar{g}_{\text{-}220} \) for their strong dark field beam image and antipodal diffraction vector, \( \bar{g}_{\text{220}} \), for the weak beam dark field image. They showed theoretical curves which strongly resolved the image of the partial dislocations. They also presented a weak beam, dark field image of a dislocation in Cu + 10 wt\%Al in which the two partial dislocations were 120A apart. By using the anisotropic elastic theory by Stroh (2), one can obtain the stacking fault energy.

Recently, Saka (3) extended this work by measuring the distance between the partial dislocations and by measuring the node size. He prepared his specimens by compressing single crystals to create dislocations. From the deformed crystals, thin segments were sliced with surfaces parallel to (111) planes. These were bent and annealed to develop nodes. From the analysis of the stacking fault energy data obtained from the distance between partial dislocations and the diameter of the nodes, it is possible to determine dislocation core cut-off radius as well as the stacking fault energy.
CONCLUSIONS AND RECOMMENDATIONS

The weak beam, dark field technique is the most powerful, direct method for determining the stacking fault energies. Implicit in the technique is the requirement for single crystal specimens. It is likely that large grain sized specimens could be used as well if care was taken in their selection and it may be possible to successfully prepare specimens from polycrystalline samples which show a strong preferred orientation.

The specimen preparation was difficult and tedious. This was particularly true for the cutting of the specimens from the single crystal. It was necessary to get the spark cutter into working condition in order to cut specimens from a previously oriented crystal.

This author brought with him a eucentric goniometer which he borrowed from the University of Missouri-Kansas City Dental School. The installation caused some delay in the project as did the failure of the high voltage transformer in the transmission electron microscope.

This project was delayed in two ways. Parts of the eucentric goniometer were not brought originally hence this delayed its installation into the microscope. The wire spark cutter had a limited supply of wire which required time to find a supplier. Soon an adequate supply of wire will be on hand. It may be that spark cutting is too damaging for the resulting specimens to be useful. To date, specimens for similar research have been prepared by abrasive wire saws.

This author is eligible for a sabbatical leave. An attempt will be made for this sabbatical leave to be taken in the academic year 1983-84 in this laboratory. If this can be arranged, the author will do preparation of specimens in his own laboratory which will accelerate the research effort.
