

AD 681359

College of Engineering
University of Washington
Seattle, Washington 98105
December 17, 1968

Final Report to Office of Naval Research

Contract Nonr - 477(40)

NR 036 061

Period Covered: June 1, 1964 - Dec. 31, 1968

THE MICROSTRUCTURAL ASPECTS OF DEFORMATION
AND FRACTURE AT ELEVATED TEMPERATURES

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by R. Taggart and D. H. Polonis

Microstructural Processes of Deformation in Metastable FCC Solid Solutions

An apparatus has been designed and constructed to permit detailed investigations to be made of the microstructural aspects of deformation in metals and alloys at elevated temperatures. It is also possible to employ this apparatus for the study of the combined effect of strain and temperature on phase transformations. Variations in the temperature, load and strain conditions can be correlated on a time basis with the resolvable microstructural changes that accompany deformation. In addition to these data it is possible to record amplified traces of the load transients which in turn can be related to the mechanisms of plastic deformation. The entire gauge section (1.5 in x 0.375 in.) of a flat tensile specimen may be surveyed and the microstructure can be recorded by photomicrography or by microcinematography. Ambient atmospheres of vacuum, argon, or helium may be interchanged at any time during an experiment. Jaw separation rates, selectable between 10^{-3} and 10^{-6} inches per second are available and are independent of the load variations. The maximum operating temperature is 1500°C, and the maximum load capability is 3000 pounds in tension.

The above apparatus has been used in a study of the strain-induced (FCC) → (HCP) phase transformation in Cu-Ge and Cu-Si alloys. The morphology of the HCP martensitic product and the effect of the HCP phase on the subsequent deformation characteristics of these alloys have been determined as a function of temperature.

Strain induced kappa phase formation has been identified as the initial deformation process that occurs in a randomly-oriented polycrystalline Cu-Si alloy. The conditions for its development within a particular grain have been determined. The unusually low yield strength observed in Cu-Si alloys is attributed to the low stress level required for K_ϵ phase formation. The strain energy required to initiate the strain-induced transformation increases with temperature up to 300°C and above this temperature the strain energy requirement rapidly diminishes due to the thermal activation. A dislocation mechanism has been proposed to explain the growth of K_ϵ platelets to microscopically observable thickness, and this model is in agreement with the observed strain energy requirement. The high strain hardening rate of the metastable alpha phase in Cu-Si alloys has been identified with the slip barriers established by the hexagonal close packed plates that form during the early stages of deformation. It has also been established that the strain induced transformation in metastable Cu-Ge alloys occurs in a similar manner.

The kinetics of the thermally activated FCC $\rightarrow$ HCP reaction in Cu-Si and Cu-Ge alloys have been studied together with the morphology and the distribution of the hexagonal phase. This diffusion controlled transformation exhibits nucleation and growth kinetics and the HCP phase is nucleated primarily from stacking faults that originate at the grain boundaries and at the incoherent twin boundaries.

A study has been made of the relationship between the measured specimen strain and the electrical resistivity of Cu-Si alloys for strain levels below 0.3%. The experiments were designed to provide a quantitative description of the conditions that correspond to the initiation of plastic flow from which a detailed model of the early stages of the strain induced phase transformation has

been developed. The electrical resistivity decreases slightly during the initial stages of loading and a maximum decrease of $0.02 - 0.04\mu$ ohm-cm has been observed at $0.07 - 0.08\%$ strain. A comparison between the resistivity-strain curve and the stress-strain diagram reveals that the resistivity decrease occurs entirely within what is normally considered to be the elastic range for a metastable Cu-Si solid solution.

A mechanism to explain the initial resistivity decrease must be consistent with two important experimental observations:

- a. the resistivity decrement is found to occur during the elastic strain period.
- b. the maximum value of the resistivity decrement decreases with decreasing solution temperature.

Friedel has discussed a model proposed by Suzuki for the stress induced motion of a stacking fault that is associated with segregation and is bounded by glissile partial dislocations. According to this model the stacking fault width decreases by a fractional amount upon the initial application of stress as one partial dislocation moves toward the other, which is stationary, within the confines of the initial Suzuki segregation. The application of increasing stress ultimately results in the escape of the glissile partials from the Suzuki segregate.

This model predicts an initial decrease in the resistivity of metals of low stacking fault energy (e.g., metastable Cu-Si alloys) during straining due to the initial decrease in the stacking fault area. This mechanism also accounts for the observation of this effect during elastic deformation since the stacking fault would return to its initial dimensions upon removal of the applied stress if the fault had not escaped the Suzuki atomsphere.

To be completely successful as a model for the initial resistivity decrease the Friedel model must also explain the sensitivity of the resistivity decrease

to variations in the solution temperature.

The stacking fault energy is reduced as the temperature is lowered in systems such as Cu-Si where the boundary separating the FCC and the two phase (FCC + HCP) regions has a negative slope. Suzuki segregation to the stacking faults, which should increase in Cu-Si alloys with decreasing solution temperature, should therefore lead to a variation in the initial stacking fault size in the retained alpha phase after cooling to room temperature. The increased Suzuki segregation at lower solution temperatures and the corresponding decrease in the total stacking fault energy lead to an increase in the energy required to close a fault. On this basis it is predicted that a decrease in the resistivity decrement will occur with decreasing solution temperature which is in accordance with the behavior observed in this investigation.

During the terminal stages of the contract period special emphasis was placed on the application of transmission electron microscopy to the study of the FCC $\rightarrow$ HCP transformation. X-ray analysis revealed that the constitution of solution treated and slowly cooled specimens of Cu-11.8 wt. % Ge was entirely FCC. Transmission electron microscopy showed that the matrix contained intrinsic stacking faults and it was observed that the partial dislocations bounding these stacking faults were distributed randomly on all four variants of the {111} planes. Stair-rod dislocations were observed and the Shockley partial dislocations were all of the type $\frac{a}{6}\langle 112 \rangle$. The nucleation of the HCP phase occurred by the propagation of stacking fault bundles through the matrix until these bundles interacted to form isolated colonies of the product phase.

Optical microscopy did not reveal the presence of the strain induced HCP phase in specimens that were quenched from the solution temperature in iced brine. The strain induced phase was not observed by selected area electron diffraction techniques but it was observed that the stacking faults were usually more widely split than for the slowly cooled specimens. When the quenched specimens were aged in the temperature range 50-200°C for times ranging from one to three hours vacancy type defects were observed in the matrix, but areas near the grain boundaries were denuded of such defects. At the lowest aging temperature only black spot defects were seen but at higher aging temperatures Frank loops were present and both isolated and linear arrays of stacking fault tetrahedra could be identified.

This program has made it possible to identify the important role assumed by stacking faults in the FCC $\rightarrow$ HCP transformation in alloys of low stacking fault energy. It has also been shown that the solution treatment prior to quenching is an important variable influencing both thermally activated and strain activated processes. The results of this program contribute to an improved understanding of the fundamentals underlying the thermo-mechanical processing of alloys.

Surface Variables and Their Effect on Deformation and Fracture

1. Liquid-Metal Embrittlement in Mercury-Zinc Couples

The effectiveness of liquid mercury as an embrittling agent when applied to zinc monocrystals was assessed on the basis of a series of deformation experiments that were carried out on single crystals grown in the shape of a tensile specimen. The advantage of a single crystal specimen with an isolated gauge length lies in the fact that the load applied through the enlarged ends of the specimen is

uniform and symmetrical with respect to the gauge section. A modified Bridgman technique was employed and the single crystal tensile specimens were produced with a random orientation between the basal plane and the tensile axis by selecting a growth rate of 1 cm. per hour and a temperature gradient of 10°C per centimeter.

It was found that liquid mercury does not affect the mechanical properties of zinc monocrystals deformed in tension provided that they have been chemically polished prior to testing. A single kink band artificially introduced into a single crystal specimen acts as a barrier to dislocation movement and is the primary cause of embrittlement over a wide range of orientations between the basal plane and the tensile axis. Artificially induced surface twins were also observed to cause the embrittlement of zinc monocrystals in the presence of liquid mercury.

Chemically polished specimens, that were cold worked by bending the gauge length prior to tensile deformation, were not totally embrittled by contact with liquid mercury. Annealing the cold worked specimens did not change their reaction to a liquid mercury environment: the zinc single crystals remained only slightly embrittled, as indicated by a small drop in the elongation prior to fracture. An acid saw cut was employed to simulate a simple notch but the resulting stress concentration did not produce extensive embrittlement in the presence of mercury.

It has been concluded from these studies that the introduction of a kink band is essential to produce embrittlement in zinc monocrystals exposed to a liquid mercury environment. This work supports the conclusions of Kamdar and Westwood regarding the initiation of fracture in zinc wetted with liquid mercury.

A fractographic study has been made of the cleavage surfaces of the zinc crystals after fracture. A rough fracture surface with many cleavage steps was typical of the mercury embrittled crystals and demonstrated clearly the reduction

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in surface energy that occurred when mercury was present on the zinc surface. If the mercury supply was depleted before the fracture process was completed it was observed that, although the crack propagation continued, the fracture surface exhibited a change in fracture mode. This effect can be explained in terms of the Griffith theory of crack propagation.

2. Liquid Metal Embrittlement in Aluminum-Gallium Couples

It has been found that when polycrystalline aluminum is wetted with gallium and the environmental temperature is varied, the fracture characteristics exhibit a brittle to ductile transition. Above the transition temperature the elongation at fracture does not approach the ductility of an unwetted aluminum specimen of identical geometry, an observation that has been attributed to the grain boundary penetration of gallium. As the strain rate is increased the ductile to brittle transition temperature is increased and the experimental results can be represented as follows: $T_c \propto \ln \dot{\epsilon}$

Fractographic examination of the specimens that failed both in the ductile and in the brittle regions indicated that the crack propagation involved the same intergranular mode in both cases.

3. Strain Distribution at the Tip of a Stationary Crack

A new experimental technique employing a pattern of microhardness indentations has been developed to permit an accurate measurement of the plastic-strain distribution at the root of a mechanically introduced notch. The method allows measurements to be made to within 0.001 in. from the crack tip.

The longitudinal strain distribution was determined for notched 2024-T3 aluminum alloy specimens, and both the longitudinal and the transverse distributions

were obtained for notched specimens of high-purity aluminum. The results from the experiments indicated that a relationship exists between the grain size and both the strain magnitude and distribution. The specimens having the largest grain size developed the highest strain gradients, and exhibited the greatest degree of symmetry about the center line. The symmetry of the strain distribution around the notch improved as the applied load approached that required to produce crack propagation whereas, at low loads, there was little evidence of any symmetry.

Effects of Environment on Surface Pit Formation in Aluminum

Vacancy condensation pits have been observed on the surface of pure aluminum after thermal cycling under an argon atmosphere. A holding temperature of 600°C or above was required. Pits formed after a ΔT of 25°C to 35°C. This is at variance with the data reported by Doherty and Davis (1959) for pit formation during cycling in air where holding temperatures below 500°C were required for maximum pit production.

The larger vacancy condensation pits formed in aluminum by quenching under an argon atmosphere were incapable of "healing" themselves upon return to the holding temperature. Calculations have shown that the thin oxide superstrate formed on the aluminum under the test conditions can collapse into the pit. Repeated thermal cycling resulted in the nucleation of small vacancy condensation pits at the bottom of many large unhealed pits, suggesting a relationship with the points of emergence of dislocations.

The experimental work has shown that when oxidation is inhibited by a vacuum treatment that results in thermal etching no pits are produced by

quenching either in vacuum or in argon. Since it has been shown that oxidation is necessary for vacancy condensation pit formation there is insufficient evidence to identify all pits with dislocation sites as has been suggested in previous studies. Furthermore, some consideration must be given to the fact that cation diffusion from the metal surface into the oxide at localized sites will provide a source of vacancies as well as an influence on pit nucleation.

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P.J. Moroz	

Unclassified

Security Classification

14	KEY WORDS	LINK A		LINK B		LINK C	
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	Mechanical Properties, phase transformations Cu-Si alloys, Cu-Ge alloys, liquid metal embrittlement, crack propagation, hot stage microscopy						

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