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EXPERIMENTS IN HIGH VOLTAGE ELECTRON MICROSCOPY

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Abstract

High voltage electron microscopy (HVEM) has been used to study radiation damage in metals and ceramics, along with supplemental investigations using neutron and ion irradiation. Studies of alloys have concentrated on radiation effects on phase stability, particularly precipitate growth and, most recently, order/disorder transitions. β-phase alloys (e.g. FeAl) disorder under irradiation, but also develop unique striated structures with streaks in the diffraction patterns (especially NiAl), which may be due to secondary defects or a second phase. In simple oxides such as MgO, Arrhenius plots of dislocation loop growth rates give a high temperature activation energy corresponding to vacancy migration and an apparent low temperature activation energy corresponding to interstitial migration. Both electron and neutron irradiation produce voids in Al_2O_3; in addition neutron-irradiated polycrystalline Al_2O_3 exhibits microcracking along grain-boundaries due to anisotropic swelling. Further studies of the radiation-induced metamictization of quartz have revealed that the strain centers which are observed prior to the general amorphization are probably amorphous inclusions; these inclusions are much more prominent in "wet" synthetic quartz than "dry" lunar quartz. Ceramics with more complex crystal structures are much more resistant to the formation of interstitial loops and voids (and hence swelling) under both electron and neutron irradiations. For example, although $MgAl_2O_4$ spinel gives rise to a low density of large interstitial loop clusters, voids are observed only near grain boundaries in polycrystalline material. In other ceramics such as YAG, Si_3N_4 and other silicon ceramics, only small defect clusters and little swelling are observed.

1. Introduction

The high voltage electron microscope (HVEM) is well established as an important research tool, not only for radiation damage studies, but also for its increased penetration of specimens, decreased aberrations and its facility for in situ experiments such as environmental interactions and deformation studies[1-3]. The research described in this report utilizes mainly the electron irradiation capabilities of the HVEM, but other aspects, especially the increased penetration, are also important.

The ability to perform radiation damage studies is important in its own right and to simulate damage produced by neutrons in fission reactors and fusion reactors and by various types of radiation in nuclear waste storage media. The HVEM can successfully simulate these damage processes at greatly accelerated rates - ~10^{-3} dpa/s for knock-on processes or ~1 dpa/s for some ionization damage mechanisms. Electron damage has the additional advantage for fundamental studies that single Frenkel pairs are produced, so that subsequent clustering behavior is more continuous and well-controlled compared with the cascade configurations produced by energetic neutrons and ions. This is particularly important for studying complex systems such as alloys and multiatomic ceramics, as in the present research. However, some supplemental comparisons of neutron/ion damage with electron damage have been performed in some of the more complex ceramic systems.

Our research over the past year has concentrated on radiation effects in alloys and ceramics. There are a variety of effects of irradiation on phase stability in alloys[4,5]; our own research has concentrated on precipitate/void/loop interactions and on effects of irradiation on precipitate growth in various alloys[6-8]. In ceramics, the response to irradiation can be even more complex due to considerations of stoichiometry and imbalance in the spectrum of primary defects (anions and cations). We have categorized these responses in three ways[8]: (a) the formation of stoichiometric defects (loops and voids), (b) decomposition or precipitation of a second phase, and (c) transition from the crystalline to the amorphous state (amorphization or metamictization).

Results of research during the past year are described below. The accounts are brief since much of the research has been, or is being, written up for publication. (See the list of Publications in Section 4).

2. Result and Discussion

2.1 Experimental Techniques

The new side-entry specimen system for the HVEM is now in routine operation. We have now added a double-tilting/cooling holder to our previous list of stages - goniometer, double-tilting/heating and environmental cell with heating and straining capability. We have also ordered a low-light TV camera for dynamic recording and are in the process of ordering a complete Analytical Electron Microscope with capabilities for high resolution TEM, STEM, SEM, XEDS and EELS. Both will be of enormous benefit to the present research. Other techniques such as ion-beam thinning, high flux irradiation, weak beam imaging and lattice fringe imaging have been described previously[10].

2.2 Radiation Effects in Alloys

Our previous emphasis has been on the study of radiation effects on precipitation and phase stability in Al- and Ni-base alloys, which have been published[6-8], and most recently in Cu-Be alloys. For the past year we have initiated a new program on order-disorder phenomena in β-phase alloys, as described below.

A method of determining the long range order parameter (S) in ordered alloys from thickness fringes in electron micrographs has been proposed by one of us[11,12]. This method has been applied to ordered Fe-49 wt. % Al (B2 type); the change of S during irradiation under HVEM follows an equation $S = S_o \exp(-kT)$, where S_o is the initial value of S, K the cross section of disordering and t the irradiation time. The values of K have been determined as a function of crystallographic orientation (α), kinetic energy of electron (E), electron flux (ϕ) and irradiation temperature (T_i). The values of K do not depend on α and are 4, 19, 48, 75, 86, and 99 barn at E = 250, 350, 500, 750, 1000 and 1250 kV, respectively [13]. Calculated disordering cross sections based on the <111> replacement collision do not agree with the observed E and α dependences of K, but calculations based on the recombination of displaced atoms with vacancies on their wrong sub-lattices agree with the ∅ and T_i dependences as well as E and α dependences of K. Furthermore, analyses show a higher value than 1 eV for the migration energy of interstitial atoms in Fe-49 at. % Al, and support the value of 1.36 eV proposed by Riviere et al.[14].

Irradiation of Fe-49 at. % Al also developes a unique striated microstructure accompanied by streaks in <110> directions[15]. Furthermore, there are a specific number of streaks at each diffraction spot and the striated structure lies perpendicular to the corresponding streaks at the particular diffraction spot which is highly excited. Tanner[16] observed similar contrast effects and diffraction patterns for Cu-2 wt. % Be alloys to those in irradiated FeAl. He pointed out that GP zones lie on {100} matrix planes but produce matrix strains in <110> directions which are primarily responsible for strain field contrast, and that the precipitate invisibility is understood in terms of a " $\vec{g} \cdot \vec{b} = o$ invisibility criterion". Here, $\vec{g}$ is the diffraction vector which is operating and $\vec{b}$ is the strain vector. This criterion explains the diffraction patterns and micrographs for FeAl containing elastic shear strains on {110} planes in <1$\bar{1}$0> directions. Fine strain contrast is also observed under weak beam conditions, and higher fluence introduces an increase in their density which leads to an amorphous-like state.

Similar observations have been made on Ni-50 at. % Al. Streaks in the diffraction patterns and striations in the micrographs are observed; they are less obvious in the unirradiated condition but become stronger during irradiation[15]. We are investigating several possible origins of these shear strain centers: (a) very fine interstitial loops, (b) very fine disordered domains, (c) fine precipitates or (d) oxides on the surfaces. Experiments at various T_i and Ø on FeAl and NiAl support (a) although some doubts still remain. Such fine interstitial loops which do not grow extensively during irradiation may be due to the high migration energy of interstitial atoms to each other, reducing the biasing factor, and, we believe, introducing an increased swelling resistance of irradiated materials.

2.3 Stoichiometric Extended Defects in Simple Oxides

Results on MgO and Al_2O_3 have been published[17-19]. Interstitial loops are formed by the aggregation of primary defects in stoichiometric proportions with perfect 1/2 <110> Burgers vectors in MgO and partial 1/3 <0001> and 1/3 $\langle 10\bar{1}0 \rangle$ Burgers vecotrs in Al_2O_3; in the latter cases the associated faults are in the cation sub-lattice only. The elongated growth mode which is observed for these loops is a result of kinetics rather than energetics, in that there is a preferred nucleation and growth of certain types of jogs in the close-packed directions. Arrhenius plots of the temperature dependence of the loop growth rates in MgO give two apparent regions: a high temperature, high activation energy region corresponding to anion vacancy migration at a low temperature, low activation energy region corresponding presumably to interstitial migration.

At high dose, voids are observed in Al_2O_3 after electron irradiation as also observed by ion and neutron irradiation[20,21]. Our results on neutron-irradiated Al_2O_3 show that the dislocation network formed prior to the voids still retains its loop character with mininal interactions[22]. The void lattice (aligned along [0001]) is also found in polycrystalline Al_2O_3; the major and important difference is that micro-cracking is evident along the grain boundaries, a result that we believe is due to isotropic swelling.

Electron irradiation studies of two other simple oxides, BeO and NiO, have just begun for the purpose of learning more about their defect migration and aggregation behavior. A startling result was obtained for NiO, in that oriented Ni precipitates with a cube/cube orientation relationship were produced by annealing and irradiation above 400°C; however, it appears that this may be due to the preferential sputtering of oxygen atoms during argon ion thinning.

2.4 Crystalline-to-Amorphous Transformation

Radiation can induce topological disorder in a crystalline solid leading to the metamict state. This is a common observation during TEM of silicate minerals, and quartz in particular has been extensively studied. However little progress had been made until recently in understanding the fundamental, as opposed to the phenomenological, aspects of the damage mechanism.

Irradiate α-quartz undergoes a crystalline → amorphous transformation involving a 14% decrease in volume. Our current investigation of this material has involved in-situ electron irradiation of hydrothermally-grown synthetic α-quartz and lunar α-quartz in our Siemens Elmiskop 102 125 kV microscope and in our Hitachi 650 kV HVEM at electron energies from 20 keV to 650 keV. Subsequent analysis has made extensive use of weak-beam dark-field imaging techniques at high magnifications ($>10^5$). Our observations have enabled us to deduce the following features of the degradation mechanisms involved.

a) The crystalline → amorphous transformation takes place in two stages at different rates. First comes inhomogeneous nucleation and subsequent growth of at least partially amorphous strain centers, and second a gradual homogeneous loss of long-range order in the remaining matrix.

b) Both damage processes scale with the ionization cross section and without apparent threshold, and are therefore attributable to radiolysis, in contrast to conclusions of previous investigators. This is particularly significant in waste storage applications involving SiO_2-based solids where a major component of the radiation field is high-energy electrons which lose most of their energy in ionization events. It is also an important piece of information for assessing the radiation hardness of quartz frequency standards. The overall metamict transformation is essentially complete after a dose of about 100 G Gy. This represents a damage efficiency of about 0.1 MeV deposited energy per bond broken. The dose to nucleate strain centers is approximately 100 times less.

c) The strain centers are three-dimensional misfitting inclusions and not dislocation loops as previously claimed by other investigators. They grow to dimensions exceeding 20 nm and are approximately spherical but early on develop facets on $\{11\bar{2}0\}$ prism planes. They appear to be partly or substantially transformed to the amorphous state (up to 14% lower density) and induce large internal stresses due to the strain of accommodating locally transformed regions in surrounding untransformed material. As irradiation proceeds, matrix strain-field contrast is lost, possibly because the matrix surrounding the strain center gradually transforms as well, lessening both misfit and diffraction contrast.

d) Our comparative study of essentially water-free lunar α-quartz reveal reveal a much lower nucleation density of strain centers than in the hydrothermally-grown synthetic quartz containing 100-1000 ppm water. We therefore suggest that weak hydrolyzed Si-OH:HO-Si bonds could serve as nucleation sites for the amorphous inclusions. Growth of the inclusions involves migration of radiation-produced point defects as evidenced by regions adjacent to boundaries, faults and interfaces denuded of such inclusions.

e) Loss of long-range order in a connected structure like SiO_2 must involve reorientation and rebonding of [SiO_4] tetrahedra which we speculate can only take place in the presence of a high local concentration of point defects providing the requisite freedom. The importance of reorientation in providing nucleation sites for extensive damage is suggested by our observations of preferentially accelerated damge occuring at Dauphiñe twin boundaries. We have therefore formulated a model for the initial radiolysis mechanism which involves coupled formation of E_1' oxygen vacancy centers by Si-O bond excitation followed by rebonding of the non-bridging oxygen to a neighboring oxygen atom in an $O_2^=$ peroxy linkage, stabilizing what is essentially a close Frenkel pair. The presence of both these point defects in irradiated SiO_2 is now certain from recent EPR measurements.

Several further experiments are in progress:

a) Examination of damage for electron energies well belwo 20 keV to confirm that the damage processes are truly radiolytic.

b) Studies of damage rate as a function of temperature from 4 K to 800 K to confirm the role of mobile point defects in the growth of strained amorphous inclusions.

c) Annealing studies of damage formed at room temperature at various stages of the transformation to ascertain its reversibility and investigate recrystallization behavior.

d) Examination of specimens with a wide range of water content to quantify the effect of hydrolyzed bonds on damage nucleation rate.

e) In-situ electron irradiations of other SiO_2 crystalline polymorphs (cristobalite, tridymite, coesite) and of GeO_2 polymorphs (trigonal, tetragonal) to investigate the structural dependence of the crystalline → amorphous transformation.

2.5 Non-swelling ceramic systems

The nuclear industry has current and projected needs for refractory insulating solids characterized by low swelling and structural integrity in severe radiation environments. Density measurements at LASL have indicated that certain complex ceramics, such as $MgAl_2O_4$ spinel, $Y_3Al_5O_{12}$ (YAG) and Si nitrides and oxynitrides, exhibit negligible swelling even to doses as high as 20 dpa in contrast to simpler ceramics such as BeO and Al_2O_3 which swell catastrophically and anisotropically.

Spinel and YAG have the advantage of being cubic and thus reasonably isotropic. We have sought evidence from TEM to explain their radiation resistance. Single crystals of stoichiometric $MgAl_2O_4$ and $Y_3Al_5O_{12}$ were neutron-irradiated under LASL auspices in EBR-II to fluences (>0.1 MeV) of 3×10^{25} nm^{-2} (~3 dpa) and 2×10^{26} nm^{-2} (~20 dpa) at temperatures between 925 K and 1100 K. Aggregated damage was not resolved in the lower-dose samples (by comparison, alumina at this dose exhibits void arrays), but the higher dose samples contained large faulted-dislocation loops. These loops were noted but not analyzed yet in YAG, as the crystallography is complex with 156 atoms/unit cell.

Spinel exhibited two types of faulted loops:

a) faulted loops of interstitial character on {110} planes with $\underline{b}$ = 1/4 <110>. Cluster of these loops were observed to grow from a central point on all six {110} planes to form rosettes. At the higher temperature, the loops had grown to such large dimensions (>1 μm) that they intersected the foil surfaces. Double layer loops were also observed which locally removed the fault, but it is clear that a facile shear mechanism for unfaulting these loops does not exist (as it does in Al_2O_3), and the loops remain rigorously planar and prismatic.

b) Faulted loops of undetermined character on {111} planes with $\underline{b}$ along <111>.

The 1/4 <110> {110} loops can be explained as condensation of two extra layers in the 110 planar stacking sequence of idealized spinel

a α a β a α | a α | a β a α a β a

where the a layers contain O and Al ions and the α and β layers contain O, Al and Mg ions (assuming no cation inversion). Insertion of the two layers | a α | preserves both the stoichiometry and the anion stacking sequence but introduces a cation fault.

The <111> {111} loops are more difficult to explain. The stacking sequence of {111} planes in idealized spinel is

$$a\ \gamma\ b\ \alpha'\ c\ \beta\ a\ \gamma'\ b\ \alpha\ c\ \beta'\ a$$

where a, b and c are oxygen layers, α, β and γ are kagome layers containing only Al, and α', β' and γ' are mixed layers containing Al and Mg. A Burgers vector of $\underline{b}$ = 1/3 <111>, corresponding to insertion (or removal) of four layers, preserves stoichiometry but introduces both a cation and an anion fault; $\underline{b}$ = 1/2 <111> produces only a cation fault but alters stoichiometry and charge balance and involves six layers; $\underline{b}$ = 1/6 <111> is the shortest Burgers vectors, involving only two layers, but introduces anion and cation faults and alters stoichiometry and charge balance. Local cation inversion is possible to correct the stoichiometry and the charge problems, but simultaneous anion and cation faults are less likely.

A recent observation in polycrystalline $MgAl_2O_4$ is that small inclusions (probably voids) form adjacent to the denuded regions surrounding grain boundaries. This presumably implies that boundaries are better biased interstitial sinks than are the faulted loops. Recombination is therefore the dominant fate of Frenkel pairs in $MgAl_2O_4$ which accounts for the low swelling rate.

We are continuing this study in spinel with additional analysis of neutron-irradiated samples and in-situ electron irradiaton in the HVEM. The latter has produced faulted loops. The possiblity of judicious choice of electron energy opens up the possibility of investigating the effects of non-stoichiometric displacements in complex ceramics since stoichiometry alterations are clearly involved in at least some of the expanded defect structures. YAG appears in TEM even more radiation resistant than $MgAl_2O_4$, but the crystallography and site multiplicity are daunting. The Yttrium ion is too heavy to be displcaed by our 650 keV electrons, and HVEM irradiation at ~1100 K has produced no visible damage, even though Al and O ions are presumably displaced. The nitrides and oxy-nitrides are intriguing but currently offer less prospect of understanding the fundamental mechanisms of defect stabilization than do YAG and spinel.

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Several other papers are in preparation.