FRACTURE TOUGHNESS OF ORDERED INTERMETALLIC COMPOUNDS
EXHIBITING LIMITED DUCTILITY AND MECHANICAL PROPERTIES OF ION-
IRRADIATED POLYCRYSTALLINE NiAl

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I. SUMMARY OF RESEARCH

The focus of the research performed under the auspices of this grant changed several times during the lifetime of the project. The initial activity was an investigation of irradiation-induced amorphization of ordered intermetallic compounds, using energetic protons as the bombarding species. The principal characterization techniques were transmission electron microscopy (TEM) and x-ray diffraction. For the first few years of the project Professor C. N. J. Wagner was a co-Principal Investigator because of his expertise in x-ray diffraction procedures. Two significant events stimulated a change of direction: 1. The proton accelerating facility that we had been using at the California State University at Los Angeles became unavailable late in 1988 because of a personnel matter involving the only individual capable of operating the machine; 2. We learned that disordering and amorphization of intermetallic compounds produced interesting effects on their mechanical properties.

Loss of access to the local accelerator prompted a collaboration with Dr. Dora Pedraza of the Oak Ridge National Laboratory (ORNL), enabling access (albeit limited) to the accelerator at ORNL. The influence of disordering and amorphization on mechanical properties ultimately stimulated the development of a miniaturized disk-bend testing (MDBT) facility, the intent of which was to provide semiquantitative and even quantitative measures of the mechanical behavior of ion-irradiated ordered intermetallic alloys. The second phase of the project involved the perfection and usage of the MDBT, and involved exploratory experiments on unirradiated materials like amorphous alloy ribbons and brittle grain boundaries in Ni_3Al .

We eventually realized that the MDBT could be used to measure the fracture toughness of brittle materials like ceramics, which exhibit no plasticity at all prior to fracture, and used the MDBT in conjunction with the controlled-flaw test to verify the method. The method was also used to measure the fracture toughness of the brittle ordered intermetallic compounds Ni_3Ge and Ti_5Si_3 . Finally, with the assistance of the finite-element program NIKE 2-D, the MDBT was employed to measure the fracture toughness of two ordered intermetallic compounds, NiAl and TiAl , which exhibit a small amount of plasticity prior to fracture. It is this last effort that is reflected in the name of the project that appears on the title page of this report.

What follows is a brief summary of the research highlights of the project, organized according to the activity that was emphasized at the time.

I.1. Amorphization and Disordering of Ion-Irradiated Ordered Intermetallic Alloys

The internal structures of amorphous $\text{Ni}_{50}\text{Ti}_{50}$ and $\text{Cu}_{50}\text{Zr}_{50}$ alloys prepared by different methods were compared using x-ray diffraction. The $\text{Ni}_{50}\text{Ti}_{50}$ alloy was amorphized using 2 MeV proton irradiation and mechanical alloying, while an additional amorphous $\text{Cu}_{50}\text{Zr}_{50}$ alloy was prepared by melt spinning. In all cases the structures were found to be identical. So far as we are aware, this was the first time such experiments had been performed.

The crystalline to amorphous (C-A) transition induced by 2 MeV proton irradiation of a NiTi alloy was investigated at low temperatures using TEM and fractography. The fractographic studies indicated that the irradiated region contains two layers, one closer to the free surface consisting of a mixture of amorphous and crystalline phases, and a deeper completely amorphous phase. The progress of the C-A transition was dependent on dose and temperature. Amorphization commenced well before the crystalline matrix was completely disordered, and the amorphous phase was generally observed to nucleate homogeneously throughout the crystalline matrix. The C-A transition induced by proton irradiation more closely resembled an ion-irradiation-induced than an electron-irradiation-induced transition. The spatially non-uniform build-up of point defects during proton and heavy-ion irradiation was invoked to explain the different responses of intermetallic compounds to ion and electron irradiation.

The ordered intermetallic compound Zr_3Al was irradiated with 3.8 MeV Zr^{3+} ions to various doses at 250 °C and the irradiation-induced microstructures were investigated by TEM. Disordering was seen at the lowest dose, 0.0033 displacements per atom (dpa), and complete loss of chemical long-range order (LRO) occurred at a dose of 0.33 dpa; the onset of amorphization was also observed at this dose. Electron diffraction patterns from irradiated samples showed satellite reflections along $\langle 011 \rangle$ in thin foils in $[100]$ orientation and streaking along $\langle 11\bar{1} \rangle$ in foils oriented $[011]$. The diffraction effects were attributed to the presence of irradiation-induced microstructural defects that, when imaged in dark field, resembled rows of dislocation loops. A model was proposed for the arrays of loops, which had Burgers vectors of the Frank type. The model accounted for the contrast effects observed in the images and the streaking and satellites seen in the diffraction patterns. At the highest dose, 1.6 dpa, a new phase, Zr_5Al_3 , appeared unexpectedly, possibly as a consequence of irradiation-induced solute segregation.

In another experiment Zr_3Al was irradiated with 2 MeV protons from -124 to 250 °C. Defects with spherically symmetric strain fields were produced at both irradiation temperatures. They were of interstitial character at -124 °C and vacancy character at 250 °C. Disorder was induced at -124 °C, whereas irradiation at 250 °C initially lowered, then slightly raised the degree of LRO above its unirradiated value at the maximum fluence. Additional defects were imaged using superlattice reflections in dark field. Small defects identified as disordered zones were also observed.

I.2. Mechanical Behavior of Ion-Irradiated Ordered Intermetallic Alloys.

Ordered Ni_3Al containing 24 at.% Al was irradiated by 2 MeV protons at -45 °C. The induced microstructural changes were investigated by TEM, X-ray diffraction, fractography and microhardness measurements. Proton irradiation produced a large volume fraction of small disordered zones in a "tweed"-type microstructure. X-ray diffraction revealed distinct splitting of the higher order fundamental peaks toward low angles, and broadening of the displaced peaks, indicating that the zones observed are small clusters of tetragonally distorted disordered Ni_3Al . The microhardness measurements suggested that the disordered zones strengthen the remaining ordered matrix considerably, most likely through a precipitation strengthening mechanism. The fractographic observations suggested that an increase in ductility accompanied disordering, as evidenced by the appearance of dimples, slip markings and river patterns on the fracture surface of the irradiated region of the sample.

The intermetallic compounds NiTi , NiTi_2 , CuZr , CuTi_2 and Zr_3Al were irradiated by 2 MeV protons at various temperatures between -175 °C and -44 °C. TEM and x-ray diffraction were used to evaluate the extents of disordering and amorphization. Both phenomena progressed to varying degrees in the five compounds, depending on the irradiation temperature and dose. It was observed that the C-A transition began before the degree of LRO was reduced significantly, and that the amorphous phases nucleated homogeneously throughout the crystalline matrices. Fractography provided a useful correlation between the features on the fracture surfaces and the microstructural alterations induced by the proton irradiations.

Because of the evident influence of irradiation-induced amorphization on mechanical behavior, a miniaturized disk-bend test (MDBT) apparatus was developed for testing specimens 3 mm in diameter and a range of thicknesses. Our apparatus incorporated several features not generally found in previous designs, including direct measurement of specimen displacement, non-critical alignment, and the

flexibility to conduct tests on clamped or unclamped samples. The original test was similar to a micro-bulge test in that the load is applied through a ball bearing 1 mm in diameter; this is the so-called ball-on-ring (BOR) mode. Displacement of the center of the specimen is measured using an LVDT, the core of which is attached to a rod maintained in contact with the specimen through the use of a weak spring. Results on four materials were presented, with sample thicknesses ranging from 25 μm to 250 μm : annealed OFHC copper, fine-grained Ni_3Al , irradiated and unirradiated Zr_3Al , and an amorphous $\text{Fe}_{80}\text{B}_{20}$ alloy ribbon. A simple procedure for measuring the contact area at yielding between the ball bearing and the specimen was described. It enabled accurate values of the yield stress to be calculated from equations for the elastic deformation of clamped specimens; previously, measurements of the yield stress from the MDBT relied on a finite element model. The accuracy was confirmed by initial tests on fine-grained specimens of Ni_3Al which were cut from the grip sections of previously tested tensile samples and containing different concentrations of Al and B. The effects of varying the clamping force on the load-displacement curves were noted, and the sensitivity of the apparatus to near-surface microstructural effects was demonstrated by comparison of the results on Zr_3Al (the irradiated samples of which were affected microstructurally only within 2 μm from the surface) and $\text{Fe}_{80}\text{B}_{20}$ (in which the loads at fracture depend on which surface of the ribbon experiences tensile loading during the test).

The mechanical behavior of Zr_3Al irradiated with 3.8 MeV Zr^{3+} ions was also investigated. The samples tested were 3 mm in diameter and either nominally 100 or 250 μm thick. They were irradiated at 250 $^{\circ}\text{C}$ to peak doses varying from 0.0041 to 2 dpa. The yield stresses, σ_y , of the thin specimens were independent of dose, within experimental error, and comparable to that of unirradiated Zr_3Al . However, for the thick specimens σ_y was significantly larger for the irradiated material, possibly increasing to a maximum at a peak dose between 0.02 and 0.06 dpa. The relative insensitivity of σ_y of the thin specimens to irradiation, compared to the thick ones, was a surprising result. Examination by TEM showed that Zr_3Al began to undergo chemical disorder at a dose as small as 0.02 dpa and was nearly completely disordered at 0.41 dpa.

Additional results of mechanical testing and TEM on the ordered intermetallic L_{12} compounds Zr_3Al , Ni_3Al , Ni_3Si and Ni_3Ge after irradiation with protons or heavy ions at high or low temperature were obtained. It was found, using the MDBT, that proton irradiation of Zr_3Al , Ni_3Al and Ni_3Si raised their yield strength; a single test on Ni_3Ge showed no effect on its fracture stress. The Vickers micro-

hardness of all four alloys was raised by proton irradiation. Irradiations caused all the alloys to disorder, the extent of which depended on the irradiation temperature. The strength increase of Zr_3Al , Ni_3Al and Ni_3Si was attributed to a combination of disordering and strengthening from defects.

The effect of ion irradiation on the microstructure and mechanical properties of Ti-52Al (TiAl) and Ti-26Al (Ti_3Al) were investigated. The alloys were irradiated with 2 MeV protons and Ar^+ ions at low temperatures (-175 to -135 °C). The yield and fracture strengths of the unirradiated and irradiated materials were determined using the MDBT. The hardness and Young's modulus of the irradiated alloys were measured using a mechanical properties microprobe. The yield strength of unirradiated TiAl and the fracture strength of unirradiated Ti_3Al were in excellent agreement with published data. The yield strength of TiAl and fracture strength of Ti_3Al increased as a result of irradiation. The strengths of both alloys were lowest for the samples irradiated to the highest Ar^+ -ion dose, but otherwise there was no correlation of strength with dose. The nanohardness of the irradiated specimens generally increased with dose, but the influence of dose on Young's modulus was erratic. Plate-shaped defects, vacancy in character, and helical dislocations were observed in irradiated TiAl by TEM. The Ar^+ -ion irradiation-induced microstructure of Ti_3Al contained defects producing mottled contrast at 1 dpa and black-spot contrast at 5 dpa. Irradiation-induced loss of long-range order was also observed in both alloys.

I.3. Investigations of Plasticity and Fracture Toughness Using the MDBT.

I.3.1. Plasticity in the MDBT

The efficacy of the MDBT was tested initially by measuring the yield strengths and ductilities of Ni_3Al alloys containing 24, 25 or 26 %Al, either boron-free or doped with 0.3 or 0.35 %B, in the BOR mode. Specimens 3 mm in diameter and approximately 200 μm thick were tested. The yield strengths were in excellent agreement with the results obtained from the uniaxial tensile tests. The load-displacement curves for the brittle alloys (all but the boron-doped 24 %Al alloy) exhibited a maximum load corresponding to crack initiation. The shapes of the deformed specimens confirmed the assumption that they deformed as if they were clamped even though they were not. The fracture surfaces of the brittle alloys were consistent with intergranular failure. Nevertheless, the ductility of the alloys increased with decreasing Al content and decreasing grain size, even for the boron-free alloys which are all brittle. The fracture stress of the boron-doped 26 %Al alloy was about 30%

greater than that of the boron-free alloy. It was argued that this is most likely a consequence of the depletion of aluminum at grain boundaries, coupled with boron segregation.

Various aspects and methods of using the MDBT were rigorously evaluated, including the relative advantages of the BOR mode and the ring-on-ring, or ROR, mode, in which the load is applied by a small (~1 mm dia) cylinder. Despite the ability to reproduce results obtained by conventional testing, several curious features were noted. For example, in tests conducted in the BOR mode correct values of the yield stress were obtained using the equations appropriate to clamped specimens, whether or not they were actually clamped in the test fixture. This appears to be ubiquitous to tests in the BOR mode, and does not arise because of frictional constraints at the supporting ring. Testing in the ROR mode was thoroughly evaluated. The yield stresses of cold-rolled or annealed AISI type 302 stainless steel were measured using various combinations of sample thickness and radii of the loading and supporting rings, and compared to those of tensile specimens machined from the same material. The most accurate and reproducible measurements of the yield strength were obtained for specific combinations of specimen thickness and geometry of the apparatus. It was demonstrated that they provide values which are always within 10% of the tensile results. The errors induced by potential misalignments in the MDBT were also discussed, and shown to cause no more than a 5% deviation in the measured yield stress. It was concluded that for quantitative measurements the ROR mode was preferable to the BOR mode, and most of the later testing was done this way.

Prior to that work, however, the MDBT in the BOR mode was used to examine the differences in the mechanical properties of the individual sides of melt-spun ribbons, only 30 μm thick, of an amorphous alloy of nominal composition $\text{Fe}_{78}\text{B}_{14}\text{Si}_8$. The curves of load vs. displacement were non-linear in nature and concave upward, rising to a peak followed by a load drop and frequently, a sustained region of unstable elongation at lower loads. Both sides of the ribbon deformed in this rather general way. The peak load was due to the start of fracture and the deflection at peak load was related to the ductility of the ribbon. The side of the ribbon in contact with the wheel (the "wheel" or "dull" side) was consistently stronger and more ductile than the side exposed to air during solidification (the "air" or "shiny" side). The wheel-side was also harder than the air-side, in agreement with previously published results. It was proposed that this behavior is due to the numerous regions of low free volume at the wheel side that are produced when air is entrapped between

the melt and the wheel, combined with local enrichment of boron and/or silicon in these same regions. These strong regions of the ribbon are interspersed with the most rapidly solidified material (having the largest free volume, highest ductility, but smallest hardness and strength), i.e. those regions that solidified while in direct contact with the wheel. The high strength and ductility of the wheel side was attributed to the heterogeneous distribution of free volume. The wheel-side was microscopically rougher than the air-side, and the anisotropy of the roughness pattern was proposed to play a significant role in the initiation of fracture.

The fracture strengths of grain boundaries in Ni_3Al were also measured using the MDBT. An ingot of directionally-solidified, boron-free Ni_3Al containing 24% Al was annealed between 1300 and 1350 °C to induce grain growth, producing many grain boundaries in excess of 1.5 mm in length. Specimens were cut from these so that one long grain boundary was located near a diameter of the specimen. The relative orientations of the grains on either side of the boundary were determined from electron channeling patterns. Low-angle boundaries were so strong they did not fracture; instead the samples deformed in a completely ductile manner. High-angle boundaries always fractured, but only after considerable plastic deformation of the two grains flanking them. Fracture was indicated by a load drop in the load vs. displacement curves. Slip traces were observed on many of the grain-boundary fracture surfaces, providing some evidence for slip transmission across high-angle boundaries. The relative fracture strengths of the high-angle boundaries were estimated by extrapolating the elastic portion of the load-displacement curves to the displacement at fracture, yielding values ranging from about 2 to 4 GPa, with an average of 3.06 ± 0.71 GPa. These are roughly an order of magnitude smaller than the fracture strengths of special boundaries predicted by computer simulations. No correlation was found between the fracture stresses and the relative orientations of the high-angle boundaries, as defined by the coincidence site lattice model.

I.3.2. Fracture in the MDBT

The MDBT, in conjunction with the controlled-flaw method, was used to measure the fracture toughness of brittle materials. The method involves the Vickers indentation of specimens ranging in thickness from 300 to 700 μm , and testing them in the ROR mode. Experiments on ZnS grown by chemical vapor deposition (CVD) were used initially to evaluate the technique. The apparent fracture toughness of this material increases with increasing crack length (R-curve behavior). The

method was then extended to measurements on other ceramic materials. Experiments on a glass-ceramic (GC) and Si_3N_4 were performed to demonstrate the validity of the technique, supplementing the original work on ZnS. The fracture resistances of these materials also increased with increasing crack length. The data were analyzed using a specific model for the relationship between fracture resistance and crack length; this model enables the R-curve behavior to be treated analytically, and the fracture resistance at "infinite" crack length to be evaluated using a straightforward graphical procedure. The resulting values of the fracture toughness for ZnS, GC and Si_3N_4 were 0.74 ± 0.02 , 2.18 ± 0.09 and $4.97 \pm 0.07 \text{ MPa}\cdot\text{m}^{1/2}$, respectively, which were all in very good agreement with values obtained from conventional fracture toughness tests on large specimens.

The fracture toughness of the brittle ordered intermetallic compounds Ni_3Ge (L_{12}) and Ti_5Si_3 were then measured, with specimens that varied in thickness from 150 to 450 μm . Ni_3Ge does not experience indentation cracking at any indentation load (unlike the previously-tested ceramics), but post-mortem examination of the fractured specimens indicated that fracture generally started at the indentation for indentation loads exceeding 38.2 N. Its fracture toughness was determined to be $7.9 \pm 1.0 \text{ MPa}\cdot\text{m}^{1/2}$. For Ti_5Si_3 indentation cracking was experienced at all indentation loads, and R-curve behavior was exhibited. Its fracture toughness was determined to be $2.69 \pm 0.21 \text{ MPa}\cdot\text{m}^{1/2}$, which is almost 30% higher than that of similar material with a larger grain size. This suggests that the fracture toughness of Ti_5Si_3 , which fractures intergranularly, might be grain-size dependent.

The method was then implemented to determine the fracture toughness of polycrystalline NiAl (grain size $\sim 25 \mu\text{m}$), which exhibits a small amount of ductility prior to failure. The specimens in this investigation ranged from 194 to 367 μm in thickness. Fracture initiated at the corners of the indentations for indentation loads exceeding 39 N. The fracture toughness was determined from an analysis of the dependence of fracture stress, σ_f , on indentation load. In brittle materials σ_f can be calculated from the measured load at fracture, but this is not possible when the specimens deform plastically prior to failure. The finite-element program NIKE2D was therefore used to calculate the stress during plastic deformation, using data on the tensile behavior of NiAl to model its deformation as an inelastic cylindrically symmetric plate. The fracture toughness of polycrystalline NiAl was measured as $6.41 \pm 1.75 \text{ MPa}\cdot\text{m}^{1/2}$, which agrees well with independently measured values for similarly processed material. The relatively large uncertainty was attributed to scatter in the experimentally measured yield stresses. The results of this investigation demon-

strate that the controlled-flaw method can be used in conjunction with the MDBT and finite-element modeling to provide a reasonable estimate of the fracture toughness of a material with limited ductility, provided fracture initiates at the corners of the indentation.

Finally, the fracture toughness of the intermetallic alloy Ti-46.5Al-2.1Cr-3.0Nb-0.2W, heat treated to produce a duplex microstructure, was measured and found to be $11.93 \pm 2.18 \text{ MPa}\cdot\text{m}^{1/2}$. This was the first time a value of fracture toughness exceeding $10 \text{ MPa}\cdot\text{m}^{1/2}$ was measured using the controlled-flaw method in conjunction with the MDBT, and the value of $11.93 \text{ MPa}\cdot\text{m}^{1/2}$ compared well with previously calculated fracture toughnesses for similar alloys.

II. PUBLICATIONS AND REPORTS

The research performed on this project resulted in the following publications. In addition to the work published in the open literature, nine annual progress reports were prepared.

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