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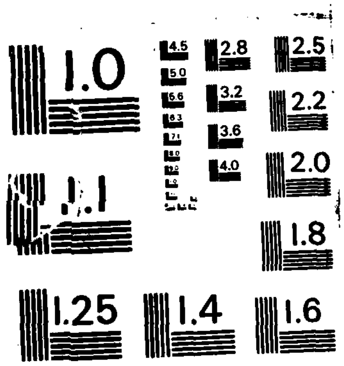


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Progress is reviewed for a research program whose primary purpose is to establish a fundamental understanding of the applications and consequences of selected advanced processing techniques to high performance alloys. The broadly-ranged research effort includes studies of powder processing (hot isostatic pressing), fracture of alloys containing processing-induced defects (porosity), and application of advanced processing techniques to high performance alloys (rapidly solidified, dispersion-strengthened titanium



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alloys). Many aspects of the program also constitute fundamental studies of deformation and fracture utilizing engineering alloys containing processing-induced defects.

Progress for the period October 1, 1985 to December 31, 1985 is reviewed for the following parts of the research program:

(1) the influence of void/pore distributions on fracture,
(2) hot isostatic pressing of metallic powders,
(3) deformation of rapidly solidified Ti alloys at elevated temperatures, and
(4) strain-path effects and the combined influence of stress state and grain size on the fracture of alloys containing hydrides.

Technical Report No. 4
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# ADVANCED PROCESSING AND PROPERTIES OF HIGH PERFORMANCE ALLOYS

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# INTRODUCTION

Advanced systems requiring high performance structural alloys which must exhibit high strength and good fracture resistance over a wide range of temperatures and environments have become increasingly materials-limited in recent years. This situation has arisen in large part because improvements in properties of such alloys have become increasingly limited to small incremental advances. At the same time, an escalation of the cost of a finished part has also confined the application of high performance alloys to selected critical components. This has resulted in an increased application of advanced processing techniques to high performance alloys <u>both</u> to create new alloys exhibiting <u>large</u> property improvements <u>and/or</u> to reduce the cost of finished component by using near net-shape fabrication methods. One example is application of rapid solidification processing as a method of developing new families of high performance alloys.[1-4] In this case, the fabrication of rapidly solidified alloys requires extensive processing based on powder metallurgy techniques. It should be noted that powder metallurgy is also being used as a new method of reducing component cost in fabricating complex-shaped components from high performance alloys.[5,6]

An underlying problem in high performance alloy technology is that the rate of applying the new processing methods to these alloys exceeds the development of the basic understanding necessary to predict the resulting component behavior in service. Thus, service reliability may be impaired. A specific concern is that these materials can contain processing-induced defects, such as porosity or inclusions, which can seriously degrade service reliablity especially with regard to fracture. Thus, the primary purpose of the present research is to provide a broad-based understanding of the applications and consequences of selected advanced processing techniques to high performance alloys. The research ranges in scope from modeling studies of the fracture of alloys containing porosity to the high temperature deformation of rapidly solidified, dispersion-strengthened Ti alloys. Much of the research is designed so that it is also a fundamental study of deformation and especially fracture utilizing engineering materials containing processing-induced defects. The present report summarizes progress in the areas of research for the period 10/1/84 to 12/31/85 performed under the auspices of Contract No. N00014-85-K-0427, NR412-019. The areas may be grouped into the following areas:

(1) the influence of voids and pores on fracture,

(2) hot isostatic pressing of metallic powders,

(3) deformation of rapidly solidified Ti alloys at elevated temperatures, and

(4) strain-path effects and the combined influence of stress state and grain size on fracture of alloys containing hydrides.

The educational experience that the above research provides to the graduate students is also significant. The following students have been supported by this program during part or all of the fifteen month period of this program: Stephen Kampe, Ph.D. candidate; Barbara Lograsso, Ph.D. candidate; Dale Gerard, Ph.D. candidate (M.S., May 1985); Paul Magnusen, Ph.D. candidate; and Ellen Dubensky, M.S. August, 1985. In addition Susan Kestner, M.S. candidate, has been supported in part by this program.

## SUMMARY OF RESEARCH

1. THE INFLUENCE OF VOIDS AND PORES ON DUCTILE FRACTURE (E. M. Dubensky, M.S., R. J. Bourcier, Ph.D., P. E. Magnusen, Ph.D. candidate).

The presence of porosity is frequently a problem in alloys which have been cast, processed by powder metallurgy techniques, or welded. In addition, wrought alloys typically contain large intermetallic phase particles which crack and form voids at small strains when fracture of the matrix is still remote. In either instance, the deleterious influence of pore and/or voids on strength and especially tensile ductility is well known (for example, see ref. 7). It is also well established that the severity of the effect depends on the volume fraction of pores or void-nucleating inclusions; however, factors such as the inclusion size and the degree of inclusion clustering should also influence fracture resistance. A difficulty in analyzing the influence of porosity or voiding on ductile fracture is that changes in one factor, for example the volume fraction of porosity, are usually accompanied by changes in other factors, such as the distributions of pore sizes, pore shapes, and interpore spacings. Thus, a separation of the <u>individual</u> effects of a pore/void microstructure on strength and fracture behavior does not exist.

The above situation is not confined to experiment; it also extends to theory. Nearly all theoretical analyses of ductile fracture based on void growth and linking assume regular arrays of equi-sized holes or cavities. Only Melander has attempted to analyze the effects of a random distribution of voids.[8,9] Little critical comparison has been made between Melander's analysis and other plastic flow models and the observed flow and fracture behavior of porous materials. The result is that the validity of the modeling techniques (especially at large strains) remains unproven, and no basis exists to suggest modifications or improvements. The purpose of this study is not only to identify the parameters which control the deformation and fracture of porous or cavitating alloys but to establish a sound physial basis, using modeling techniques, for the theory of ductile fracture of materials containing voids or pores.

In the first phase of this study, the influence of porosity on the deformation and fracture behavior of two alloys, powder-fabricated Ti and Ti-6Al-4V, with differing levels of matrix strain hardening has been examined both experimentally and analytically[10]. A large strain elastoplastic finite elment model based on a regular array of equal-sized spherical voids was used to predict bulk porosity effects. This analysis is in good agreement with the experimentally observed rates of void growth but underestimates the degradation of strength with increasing porosity. The effets of porosity on a local scale, especially as regards to fracture, have been examined by a model of a porous continuum which contains "imperfections" whose magnitude depends upon the maximum porosity path within the continuum. At critical values of strain these imperfections cause localization of plastic flow. The predicted values for the strains at localization were in good agreement with measured fracture strains. The analysis thus explicitly recognizes that a primary effect of pores on fracture is to localize deformation into narrow regions of high porosity ("imperfections") which are the sites of macrofracture initiation. Thus, the distribution of voids or pores is seen as a critical factor in determining fracture.

Many previous studies, both experimental and theoretical, have used through-thickness holes as a two-dimensional analog of a three-dimensional distribution of voids or pores. In a study in progress[11,12], we have modeled void distributions in two dimensions as arrays of holes whose positions are predicted using a random number generator. A principal result of this study is the ability to separate the individual effects of void/pore distributions on ductile fracture. Experiments have been conducted on specimens containing arrays of equi-sized holes for which the fracture behavior is monitored. The study has been based on four materials (1100 Al, 7075-T6 Al, alpha brass, and low carbon steel) of differing work-hardening and strain-rate behavior which are tested under conditions of either plane stress or plane stain as a function of (a) area fraction of holes, (b) minimum spacing between adjacent holes, and (c) size of holes.

The experimental observations in the present study lead to the following conclusions:[11,12]

1. The value of the minimum hole spacing, $S_m$, which indicates the degree of clustering among holes, has a strong influence on both ductility and yield/tensile strength. If clustering is inhibited by increasing $S_m$, ductility as well as strength increase.

2. The diameter of the holes also has the effect that decreases in both ductility (elongation to fracture) and stength (yield and tensile stengths) occur when hole size is increased.

3. The decrease in tensile strength with increasing area fraction of holes occurs in a manner consistent with data for porous P/M alloys. However, the dependence of ductility on the area fraction of holes <u>by itself</u>, is not as pronounced as porosity-effect data from bulk specimens.

4. Hole extension in the tensile direction occurs at a rate which is linear with strain. These data also show an eventual strain-induced accelerated hole growth among some but not all of the holes in the fracture path.

5. Macroscopic observations show that, upon deformation, strain localizes along a plane of maximum shear stress between either a pair or groups of holes which are both closely spaced and properly oriented with respect to the stress axis. This cluster of holes eventually forms the fracture path as the flow localization causes ligaments to fail.

6. Specimens with regular arrays of holes exhibit three to four times more ducility than those containing random arrays for the same hole size and area fraction.

The above conclusions are the basis of the following proposed sequence for the flow and fracture of materials containing voids or pores.

I. Slip initiates near individual holes (or voids/pores) as plastic zones form.

II. Flow is localized on planes of high shear stress between closely spaced and favorably oriented holes.

III. An individual ligament between two holes fractures and creates a large elliptical hole which, due to its increased size and eccentric shape, tends to further localize flow along its major axis. This causes a "percolation" effect as the increased plastic zone is more able to localize flow and cause linking with adjacent holes/voids.

IV. A statistical problem arises as to whether or not a third hole exists in a favorable position with respect to the large elliptical cavity. If a third hole is favorably located, successive flow localization and ligament fractue occurs, but if not, deformation of the entire material continues until another pair of holes/voids link up by ligament fracture, re-initiating the above sequence.

V. A group of holes (voids/pores) which have linked up cause a large imperfection. This in turn causes further localization and subsequently a shear instability over a much larger scale and finally, the material fractures.

The above sequence is sensitive to (a) minimum spacing between holes (which strongly affects shear localization between holes), (b) hole size (which controls plastic zone size and therefore the physical scale of the shear localization possible), (c) strain hardening (which tends to diffuse localization and delay the shear instabilities) and (d) in a material containing porosity or microvoids, the area fraction of pores/voids (which affects the strain necessary to link sufficient voids/pores to create the critical imperfection).

The focus of current work is to develop a theoretical basis for predicting failure based on the plastic zone geometry around holes, the statistical probability of plastic zone overlap, the plasticity around linked-hole pairs, and on imperfection theory to describe macroscopic fracture. Working with Prof. J. K. Lee of Michigan Tech and Dr. D. Srolovitz of Los Alamos National Laboratory, we are currently developing expressions which describe the zones of intense plsticity as a function of (a) hole/void shape and orientation (which has important implications to inclusion shape control and fracture), (b) average matrix strain (remote from holes/voids), (c) matrix strain-hardening exponent, and (d) state of stress. A second critical aspect is to obtain the distribution function which describes the probablility of the nearest hole/void being located a given radial distance from the reference hole/void. Once expressions are obtained for local plasticity and hole/void distribution, the problem of adjacent hole linking, percolation, and final instability will be tackled. Failure is associated with the "final instability" which in turn is a result of void linking causing a band of weakness which creates an imperfection whose severity increases with strain.

The above analysis should provide a new, statistically-based theory of ductile fracture in which the role of void/pore (and inclusion) size, spacing, and volume fraction can be predicted. Guidelines from improved properties arising from processing control are evident. For example, void-initiation sites should be controlled such that, at a given volume fraction, size is minimized, clustering is avoided, and shape is spherical. Furthermore, final fracture is seen to be an instability triggered by failure of only a few percent (or less) of a load-bearing cross section. The void linking process within most of the fracture surface is therefore a consequence of the instability. Thus analyses of fracture should focus on the conditions which cause the instability and not its consequence; fracture surface analyses, whether by fractals or surface roughness, are probably of limited value in ductile fracture.

2. <u>HOT ISOSTATIC PRESSING OF METALLIC POWDERS</u> (with Barbara Lograsso, Ph.D. Candidate)

As a means of eliminating the deleterious effects of porosity, hot isostataic pressing (HIP) is being used as a means of compacting powders, rapidly solidified ribbons, and castings to full density. In the absence of fundamental studies of HIP, unnecessarily high temperatures and pressures (plus long times) are commonly used to insure full density. For starting materials (powders/ribbons) processed by rapid solidificatiaon, this has the potential of eliminating many of the advantages inherent in the starting materials. Recently, a theoretical model[13,14] has been proposed to predict densification on the basis of creep and plastic deformation mechanisms during hot isostatic pressing. The purpose of this study has been to provide an experimental basis which established the pressure-time-temperature relationships which characterize HIP as a function of (a) particle size and (b) particle shape. The data are being compared with theory[13,14] not only as a test of its validity but also as a basis of extension.

The experimental HIP behavior of monosized spherical powders of Ni, Ti, Ti-6Al-4V, and 316 stainless steel have been examined. For the HIP conditions used (24-100 MPa pressure, $700^{\circ}$-$900^{\circ}$C, and $3\times10^{2}$ to $3\times10^{4}$s), these data show generally good agreement with theory. In addition, tests on spherical Ti powders show that powders of differnt sizes and mixtures of two sizes HIP at similar rates. Thus, absolute size and size distribution does not appear to be an important factor in HIP, which is consistent with theory. Current research is examining the effects of powder particle shape (spherical vs. irregular) on HIP behavior. Preliminary data indicate that particle shapes influence the early stages of HIP but not the latter stages. These effects are currently being related to theory using quantitative metallography of pore shapes and distributions.

## 3. DEFORMATION OF RAPIDLY SOLIDIFIED TITANIUM ALLOYS AT HIGH TEMPERATURES

(with Steve Kampe, Ph.D. candidate)

Rapid solidification (RS) represents a novel processing strategy which is currently being explored as a means to significantly increase the service temperature capability of titanium-base alloys for high performance applications. A large extension of the use of titanium alloys to higher temperatures (e.g. > 700$^{o}$C) is virtually a requirement if large improvements in gas turbine efficiency are to obtained. For example, even if structural ceramics were available such that a dramatic increase in turbine inlet temperataure were possible, concurrent increases in compressor performance (Ti alloys) is needed for overall system potential to be realized.

Despite considerable alloy development efforts directed to the development of a new generation of Ti alloys using rapid solidification (RS) processing, there has been no thorough study of the high temperature deformation and fracture behavior of RS Ti alloys in bulk form. Published research is confined to but a few tests[15], and these indicate that while some strengthening occcurs due to oxide-induced dispersion hardening, the retention of strengthening to high temperatures may not be as good as expected. This appears to be the case for several experimental alloys being developed.[16]

The purpose of this proposed project is to study in a fundamental manner the influence of temperature on the deformation and fracture of systems which model potential high temperature RS Ti alloys. The alloys, which are based on erbium additions, rely on oxide-dispersion strengthening combined with other sources of strengthening, such as void solution hardening, to achieve good creep resistance. The model alloys consist of oxides dispersed in pure alpha-phase matrix (CP Ti), solid-solution strengthened alpha-phase matrix (Ti-6Al), and age-hardened plus solid solution strengthened alpha-phase matrix (Ti-10Al and Ti-12Al).

Data from slow strain-rate compression testing of Ti and Ti-2Er as well as Ti-6Al and Ti-6Al-2Er with similar grain sizes show a definite beneficial influence of the dispersoids on the creep resistance at high stresses and low to intermediate (500°C) temperatures. However a significant loss of stengthening occurs in the dispersion hardened alloys at high temperatures(> 700°C) and low strain rates. The low values of the associated stress exponents suggest that extensive grain boundary sliding occurs in the dispersion strengthened alloys due to retention of a fine grain size by the dispersoids. Recent surface roughness measurements tend to confirm this hypothesis.

4. STRAIN-PATH EFFECTS AND THE COMBINED INFLUENCE OF STRESS STATE AND GRAIN SIZE ON THE FRACTURE OF ALLOYS CONTAINING HYDRIDES (Dale Gerard, M.S., and Susan Kestner, M.S., candidate)

a) Stress State/Grain Size Effects

Both commercially pure titanium and zirconium are resistant to embrittlement due to hydrogen when tested in the form of fine-grained specimens at low-to-moderate strain rates in uniaxial tension. However it has been shown that both Ti and Zr become more susceptible to hydrogen embrittlement at large grain sizes.[17-22] Previously we have also shown a sensitivity to stress state with Ti and Zircaloy-2 sheets exhibiting pronounced hydrogen embrittlement in equibiaxial tension due to the presence of hydrides.[23,24] The purpose of this project was to explore the combined effects of two of the above factors, grain size and stress state, on the hydrogen embritttlement of Ti.[25]

The combined influence of grain size and stress state on the hydrogen embrittlement of Ti sheet shows that there is very little, if any, effect of grain size on the ratio of fracture strain in equibiaxial tension to that in uniaxial tension for the uncharged Ti. In contrast, the loss of ductility in equibiaxial tenion for the hydrogen-charged Ti is much more pronounced for the coarse-grain material than in the finer grained counterparts. Thus, the hydrogen embrittlement of Ti sheet at room temperature is most severe in coarse-grain material when subjected to equibiaxial tension.

The above behavior may be understood on the following basis. Increasing grain size increases the susceptibility of Ti (and probably other hydride-forming alloys) to hydrogen embrittlement at high degrees of stress biaxiality/triaxiality. This effect appears to be a consequence of an enhancement of both void nucleation (due to hydride fracture) and void link-up at large grain sizes/biaxial stresses. Void nucleation should also be enhanced as the large grains create conditions for large hydrides[18,19] which in turn fracture and form voids at smaller strains at the large normal stresses required to deform plastically anisotropic (R and P>1) sheets in equibiaxial tension.[23] Void link-up will also be enhanced as the large interconnected and plate-like hydrides create paths for especially easy void link-up when subjected to the multidirectional major principal stesses in equibiaxial tension. These effects should be even more pronounced under triaxial states of stress, such as near notches or cracks, provided that the stress state is sufficiently long range to encompass several large grains/hydrides, thus permitting enhancement of both void nucleation and link-up.

b) Strain-Path Effects on Fracture

The effect of multi-stage, nonproportional strain paths on the flow behavior of metals has been examined by several investigators. In contrast, there has been no study in which the fracture behavior is examined for a metal subjected to a sequence of straining operations, each characterized by a different strain path in which the major principal strain component is tensile. If significant strain path effects occur, especially if ductility enhancement can be induced, it might be possible to design processing techniques to take advantage of them. In addition, such a combination of strain paths is of particular interest since void nucleation, void growth and void link-up are all primarily a result of tensile strains, at least on a local scale. Given the sensitivity of each stage of the ductile fracture process to a particular tensile strain path and the possibility of interactive effects, this investigation examines the ductility of a metal subjected to nonproportional, multi-stage strain paths including combinations of (a) uniaxial tension, (b) plane-strain tension, and (c) equibiaxial tension. The study utilizes titanium at two levels of hydrogen (30 and 650 wppm) so that tests may be performed on a material which readily forms voids (Ti-650 H) in comparison to that which does not (Ti-30 H).

The experimental aspects of the study are based nonproportional strain paths achieved by employing a two-stage deformation operation based on (a) a pre-strain operation in either uniaxial tension or equibiaxial tension, and (b) a final strain operation of uniaxial tension, plane-strain tension or equibiaxial tension. To assess the magnitude of the pre-strain and final fracture strains, local strain data has been obtained by the measurement of a fine grid pattern ($\simeq$ 1 mm) which is applied to the sheet surface prior to mechanical testing.

A significant observation of this study is that there is a dependence of ductility on multi-strain path. The effect is sensitive to the relative magnitudes of the multi-stage strain increments and is most pronounced at a final strain state of plane-strain tension. Specifically, the maximum ductility enhancement occurs for a combination of two nonproportional paths (i.e., uniaxial equibiaxial tension as well as equibiaxial uniaxial tension) and under conditions which result in a final strain state of plane-strain tension. Hydrogen acts to reduce the ductility enhancement. This effect can be understood by considering the influence of stress state on the ductile fracture processes. Using an analyses for void nucleation based on the maximum principal stress and given the anisotropy of Ti, it is recognized that the proportional loading in plane-strain tension occurs under the largest imposed maximum principal stress. As a result, the proportional strain path of plane-strain tension is especially effective in the nucleation of voids at small strains independent of hydrogen level. The effective nucleation of voids initiates the ductile fracture process and thus limits the ductility for the proportional strain path of plane-strain tension. In contrast, for the nonproportional strain paths in which the ductility enhancement was achieved, the material was not subjected to the large normal stress characteristic of plane-strain tension. Therefore, a ducility enhacement can be obtained by delaying void nucleation.

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3. "Advanced Processing and Properties of High Performance Alloys", Technical Report No. 3, November, 1985.

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1. "Void/Pore Distributions and Ductile Fracture of Aluminum Alloys" (with E. M. Dubensky), to be published in Proc. Int. Conf. on Aluminum Alloys, Charlottesville, VA, June 1986.

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