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Titanium alloys are attractive structural materials because of their high strength to weight ratio and excellent corrosion resistance. Alloys such as Ti-6242 and Ti-6A1-4V are used extensively in aero-engine and aerospace applications, as well as in the biomedical field due to their relatively good bio-compatibility.

In spite of their high strength, it has been recognized for some time that Ti alloys must be employed conservatively because o their tendency to creep significantly at room temperature, even at stresses well below the macroscopic yield strength. In this AFOSR funded program, we have developed an improved phenomenological and mechanistic understanding of room temperature creep.

Primary creep is the dominant mode of deformation for titanium alloys at lower temperatures under most service conditions. Significant levels of creep strain may occur at stresses, which are well below the yield strength of these alloys. The goals of this research program have been to ascertain the mechanisms, which contribute to primary creep at lower temperatures, and t determine the influence of the prominent microstructural elements on primary creep in these alloys.

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MICROSTRUCTURE AND MECHANISTIC STUDY OF CREEP IN TITANIUM ALLOYS

FINAL REPORT
AFOSR GRANT # F49620-98-1-0391

Submitted to:

Metallic Structural Materials Program
Air Force Office of Scientific Research
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Washington, D.C.
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April 2002

1. BACKGROUND

Titanium alloys are attractive structural materials because of their high strength to weight ratio and excellent corrosion resistance. Alloys such as Ti-6242 and Ti-6Al-4V are used extensively in aero-engine and aerospace applications, as well as in the biomedical field due to their relatively good bio-compatibility.

In spite of their high strength, it has been recognized for some time that Ti alloys must be employed conservatively because of their tendency to creep significantly at room temperature, even at stresses well below the macroscopic yield strength. In this AFOSR-funded program, we have developed an improved phenomenological and mechanistic understanding of room temperature creep.

Primary creep is the dominant mode of deformation for titanium alloys at lower temperatures under most service conditions. Significant levels of creep strain may occur at stresses which are well below the yield strength of these alloys. The goals of this research program have been to ascertain the mechanisms which contribute to primary creep at lower temperatures and to determine the influence of the prominent microstructural elements on primary creep in these alloys.

Titanium alloys are widely used in structural applications in which benefit can be derived from their high strength, low density and corrosion resistance. A significant limitation in the use of Ti alloys that have a large volume fraction of the alpha (hcp) phase is creep at lower temperatures. The so-called "cold creep" phenomenon has been recognized for over thirty years [1-5]. Its characteristics include: (a) Development of significant accumulated strains, even at stresses well below the macroscopic yield strength [2-5]. (b) The primary transient generally follows a power-law in time: $\varepsilon = At^a$, where A is stress-dependent [5-6]. (c) The magnitude of accumulated strain depends on microstructure in α/β alloys: larger slip distances tends to result in larger creep strains.

2. SUMMARY OF RESULTS

During this AFOSR program, we have made important strides in further characterizing this creep behavior and identifying the controlling mechanisms. This work constitutes three PhD. Theses: Neeraj Thirumalai (7/00 The Ohio State University), Satyarth Suri (7/00 The Ohio State University), and Michael F. Savage (10/00 The Ohio State University). The important findings from this work are briefly summarized below, along with publications related to each:

(1) On a phenomenlogical level, the large creep strains are associated with the extraordinarily small strain hardening rates in these alloys:

- S. Suri, D.-H. Hou, *T. Neeraj*, G. S. Daehn, J. M. Scott, R. W. Hayes and M. J. Mills, "Creep of Titanium Alloys at Lower Temperatures", *Mater. Sci. and Eng. A*, vol. A234-236, 996 (1997).

- T. Neeraj, D.-H. Hou, G. S. Daehn and M. J. Mills, "Phenomenological and Microstructural Analysis of Room Temperature Creep in Titanium Alloys," *Acta Mater.*, vol. 48, p. 1225 (2000).

(2) A cellular automata model has been developed which includes the local interactions between neighboring microstructural regions without intrinsic hardening. This model has been shown to provide a very general explanation for the observation of creep transients which obey power-laws in time, as in the case of room temperature creep in Ti alloys:

- T. Neeraj, M. F. Savage, M. J. Mills and G. S. Daehn, "Primary Creep in Polycrystalline Alpha Titanium Alloys: Coupled Observations and Stochastic Cellular Automaton Model, *Creep and Fracture of Engineering Materials and Structures*, ed. J. D. Parker (Institute of Metals, London, 2001), p. 49.

- G. S. Daehn, "Modeling Thermally Activated-Deformation with a Variety of Obstacles and Its Application to Creep Transients," *Acta Mater.* **49**, pp. 2017-2026 (2001).

(3) The presence of short-range order (SRO) in Ti-6wt%Al has been confirmed for the first time by neutron diffraction:

- T. Neeraj, J. L. Robertson and M. J. Mills, "Characterization of Short-Range Order in Ti-6wt%Al," *Phil. Mag. A.*, in preparation.

(4) The effect of heat treatment on SRO state and creep response has been examined for the first time. It was found that SRO provides significant strengthening and that the creep transients are significantly altered due to changes in the SRO state. Enhancement of SRO leads to improved creep strength:

- T. Neeraj and M. J. Mills, "Short-Range Order and Its Effect on the Primary Creep Behavior of a Ti-6wt%Al Alloy," *Mater. Sci. Eng. A*, 319-321, pp. 415-419 (2001).

- T. Neeraj and M. J. Mills, "Estimation of Strengthening Due to SRO Based on TEM Observations," *Phil. Mag. A*, in preparation.

- T. Neeraj, G. S. Daehn and M. J. Mills, "Effect of Short-Range Order on the Room Temperature Creep Behavior of Ti-6wt%Al," *Acta Mater.*, in preparation.

(5) Weak-fringing faults associated with the planar slip bands in the α-phase have been thoroughly characterized using TEM. The faults correspond to a small residual displacement of the order of ***b***/40 for prism slip bands, where ***b*** is the a/3<11-20> Burgers vector of the matrix. These residual faults are attributed to the presence of SRO in the a-phase:

- T. Neeraj and M. J. Mills, "Weak-Fringing Faults in Ti-6wt%Al," *Phil. Mag. A.*, **82**, pp. 779-802 (2001).

(6) Single colony crystals of Ti-5Al-2.5Sn and Ti-6242 have been determined to deform anisotropically with respect to the operative slip system in the α-phase. This anisotropy is related to the ease with which slip can be transmitted across the β-laths in the colony structures:

- S. Suri, D.-H. Hou, T. Neeraj, J. M. Scott, and M. J. Mills, "Effect of α/β Interfaces on the Creep Behavior of Single Colony Titanium Alloys at Room Temperature", *Interfacial Engineering for Optimized Properties*, edited by C. L. Bryant and C. B. Carter (Materials Research Society, 1997), 458, p. 267.

- M. J. Mills D. H. Hou, S. Suri and G. B. Viswanathan, "Orientation Relationship and Structure of α/β Interfaces in Conventional Titanium Alloys," *Boundaries and Interfaces in Materials: The David A. Smith Symposium*, eds. R. C. Pond, W. A. T. Clark, A. H. King and D. B. Williams, (TMS Publications, Warrendale, 1998), p. 295.

- M. F. Savage, J. Tatalovich, M. D. Uchic, M. Zupan, K. J. Hemker and M. J. Mills, "Deformation Mechanisms and Microtensile Behavior of Single Colony Ti-6242Si"*Mater. Sci. Eng. A*, 319-321, pp. 398-403 (2001).

(7) Extensive TEM investigation has revealed the mechanisms of slip transmission in several different orientations, which provide insight into the observed anisotropy in yield and creep behavior. Qualitatively these results can be explained on the basis of the Burgers orientation relationship which exists in these alloys:

- S. Suri, G. B. Viswanathan, T. Neeraj and M. J. Mills, "Room Temperature Deformation and Microstructural Characterization of Two Phase α/β Titanium Alloys ," *Acta.Mater.*, vol. 47, pp. 1019-1034 (1999).

- M. F. Savage, J. Tatalovich and M. J. Mills, " Mechanisms of Slip Transmission at α/β Interfaces and the Anisotropic Plastic Response of Ti-6242 Colony Crystals," *Phil Mag. A*, submitted April 2002.

(8) Tension/compression asymmetry (henceforth referred to as T/C asymmetry) has been observed in polycrystalline Ti-6Al (single phase α alloy) and Ti-6242(two-phase α/β alloy). Under a stress corresponding to the yield strength in tension, the strain accumulation is remarkably larger for tensile versus compressive loading. A similar behavior is observed for single-phase alpha Ti-6Al. A crucial new piece of evidence regarding the source of the T/C asymmetry is offered by the yield strength data obtained using micro-tensile and compression experiments on single α/β colony crystals of two different alloys, Ti-6242 and Ti-5Al-2.5Sn-0.5Fe. In all of these tests, the samples were oriented in order to activate a *single* a-type basal slip system. The specimens contained a single α/β colony variant, and were grown in collaboration with Wright Laboratories and Drs. Mike Uchic and Dennis Dimiduk. Significantly larger yield strengths are measured in tension versus compression. These results strongly suggest that the T/C asymmetry is an intrinsic property of a-type slip.

- T. Neeraj, M. F. Savage, J. Tatalovich, G. B. Viswanathan and M. J. Mills, "Tension-Compression Asymmetry During Low Temperature Creep of Ti Alloys," *Acta Mater.*, in preparation.

(9) A second anomalous characteristic of room temperature creep which we have observed in Ti alloys is the remarkable recovery of strain hardening while in the unloaded state. Consider a sample that has been crept to some strain value, unloaded for some period of time, then reloaded at the initial stress level. If recovery occurs during the unloading, then the

creep rate following the reload will be larger than that prior to the unload. Such a recovery process is not unexpected if the material is at moderate or high homologous temperatures since diffusion-assisted processes can lead to a reduction in stored strain energy and dislocation density. More unexpected is our observation of a truly dramatic recovery of strain hardening during static annealing at room temperature ($0.15T_m$) for several different types of Ti alloy microstructures. Based on the present, limited investigation of this effect, the change in the strain rate appears to be a function of several parameters, including duration of time unloaded and strength of the SRO state. In addition, testing of single colony crystals has revealed that certain colony orientations are particularly susceptible to the recovery phenomenon. One possibility to explain these results is that new, fresh dislocation sources are generated during the unloading period. Source generation may be driven by the large local stresses which develop at grain and interphase boundaries during planar slip. The remarkable aspect of this recovery behavior is that the accumulated creep strains following an unloading/reloading sequence can be significantly larger than would have accumulated under monotonic conditions (for the same total elapsed time).

Conclusions

Titanium alloys are utilized extensively in critical aeroengine components, including disks and blades, and hence is of significant relevance to present and future Air Force systems. Macroscopic dimensional changes due to creep and cyclic loading conditions are critical for design and component lifing considerations. Our studies have focused on the dislocation-level processes associated with room temperature creep, the effect of short-range ordering on the response, and the anisotropy in creep as a function of colony crystal orientation. In addition, this work has revealed two other remarkable characteristics of ambient temperature creep: (a) a marked tension/compression asymmetry and (b) dramatic recovery of strain hardening at room temperature. Complete characterization of these latter two characteristics is part of ongoing research funded by AFOSR in the renewal of this program.

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5. T. Neeraj, D.-H. Hou, G. S. Daehn and M. J. Mills, "Phenomenological and Microstructural Analysis of Room Temperature Creep in Titanium Alloys," *Acta Mater.*, vol. 48, p. 1225 (2000).
6. H. P. Chu, *Journal of Materials*, 1970, **5**, 633.

3. PERSONNEL SUPPORTED UNDER PROGRAM

Faculty:

Professor Michael J. Mills	Principal Investigator
Professor Glenn S. Daehn	Co-PI

Post Doctoral Associates:

Dr. G. Babu Viswanathan	SEM, TEM and spectroscopic analyses (1998-2000)
Dr. Neeraj Thirumalai	Mechanical testing and TEM analysis (8/00-7/01)

Graduate Research Associates (all at The Ohio State University):

Neeraj Thirumalai	PhD. Awarded 7/00
Satyarth Suri	PhD. Awarded 7/00
Michael F. Savage	PhD. Awarded 10/00
Matthew Brandes	PhD. Student (06/01-present)
Junho Moon	PhD. Student (09/01-present)

Undergraduate Research Assistants:

Jonathan Kline	Metallography, machining, annealing studies (1998-1999)

4. PUBLICATIONS

Publications in Peer-Reviewed Journals:

S. Suri, D.-H. Hou, *T. Neeraj*, G. S. Daehn, J. M. Scott, R. W. Hayes and M. J. Mills, "Creep of Titanium Alloys at Lower Temperatures", *Mater. Sci. and Eng. A*, vol. A234-236, 996 (1997).

S. Suri, G. B. Viswanathan, T. Neeraj and M. J. Mills, "Room Temperature Deformation and Microstructural Characterization of Two Phase α/β Titanium Alloys ," *Acta.Mater.*, vol. 47, pp. 1019-1034 (1999).

T. Neeraj, D.-H. Hou, G. S. Daehn and M. J. Mills, "Phenomenological and Microstructural Analysis of Room Temperature Creep in Titanium Alloys," *Acta Mater.*, vol. 48, p. 1225 (2000).

M. F. Savage, J. Tatalovich, M. D. Uchic, M. Zupan, K. J. Hemker and M. J. Mills, "Deformation Mechanisms and Microtensile Behavior of Single Colony Ti-6242Si" *Mater. Sci. Eng. A*, 319-321, pp. 398-403 (2001).

T. Neeraj and M. J. Mills, "Short-Range Order (SRO) and Its Effect on the Primary Creep Behavior of a Ti-6wt% Al Alloy", *Mater. Sci. Eng. A*, 319-321, pp. 415-419 (2001).

T. Neeraj and M. J. Mills, "Weak-Fringing Faults in Ti-6wt%Al," *Phil. Mag. A.*, **82**, pp. 779-802 (2001).

M. F. Savage, T. Neeraj, G. S. Daehn and M. J. Mills, "Observation of Room Temperature Recovery During Creep of Titanium Alloys," *Metall. Trans. A*, **33**, p. 891 (2002).

Publications in Conference Proceedings:

S. Suri, D.-H. Hou, *T. Neeraj*, J. M. Scott, and M. J. Mills, "Effect of α/β Interfaces on the Creep Behavior of Single Colony Titanium Alloys at Room Temperature", *Interfacial Engineering for Optimized Properties*, edited by C. L. Bryant and C. B. Carter (Materials Research Society, 1997), 458, p. 267.

S. Suri, *T. Neeraj*, G. S. Daehn, D.-H. Hou, J. M. Scott, R. W. Hayes and M. J. Mills, "Mechanisms of Primary Creep in Titanium Alloys at Lower Temperatures", *Creep and Fracture of Engineering Materials and Structures*, edited by J. C. Earthman and F. A. Mohamed, (TMS Publications, Warrendale, 1997) p. 119.

G. S. Daehn, G. B. Viswanathan, M. J. Mills and V. K. Vasudevan, "A Model of Creep Based on Interacting Elements and Its Application to the Primary Creep of Fully-Lamellar γ-TiAl", *Creep of Alloys for the 21st Century,* (TMS Publications, 1999), pp. 31-40.

T. Neeraj, M. F. Savage, M. J. Mills and G. S. Daehn, "Primary Creep in Polycrystalline Alpha Titanium Alloys: Coupled Observations and Stochastic Cellular Automaton Model, *Creep and Fracture of Engineering Materials and Structures*, ed. J. D. Parker (Institute of Metals, London, 2001), p. 49.

Chapters in Books:

M. J. Mills and T. Neeraj, "Dislocations in Metals and Metallic Alloys," Encyclopedia of Materials Science and Technology, edited by K. H. J. Buschow, R. W. Cahn, M. C. Flemings, B. Ilschner, E. J. Kramer and S. Mahajan, Elsevier Science Ltd. , 2001, pp. 2278-2291.

6. INTERACTIONS / TRANSITIONS

- Phenomenological aspects of transient creep, and its dependence on crystal orientation, are being used to aide in the development of microstructurally-based models for dwell fatigue in a FAA-sponsored program, and directly involves Andy Woodfield (GE), Jack Schirra (Pratt-Whitney) and Amit Chatterjee (Rolls-Royce).

- Extensive interaction with Dr. Lee Semiatin at AFWRL for development of models for thermo-mechanical processing of Ti alloys.

- Discussions with Jack Schirra (Pratt-Whitney) concerning Ni effects on creep in Ti alloys.

7. NEW DISCOVERIES

- The minimization or elimination of short-range ordering in the α-phase of titanium alloys is possible through heat treatment and quenching from high in the α-phase field. This modification of SRO results in dramatically different microstructures and offers the potential for improved creep, strain hardening and fatigue properties in Ti alloys at lower temperatures. The loss of strengthening due to SRO must be compensated by an increase in solute or other strengthening mechanism I order for the approach to be of practical value.

- A dramatic difference in low temperature creep response has been observed in several Ti alloys when tested in tension versus compression, the former being notably weaker. This asymmetry in response is not presently taken into account in modeling of properties in Ti alloys.

- Several Ti alloys exhibit a significant softening during extended timers an unloaded condition. Increase in the creep rates by several orders of magnitude is possible, and the effect appears to depend on time while unloaded and SRO state. This anomalous effect is also not generally taken into account in property modeling of Ti alloys.

8. HONORS / AWARDS

- M. J. Mills has been awarded the American Society for Metals Silver Medal for Research for 1998. This award was established in 1986 to "recognize and honor an active researcher whose individual and collaborative work has had a major impact on the field of Materials Science. It is intended for someone near mid-career (about 5-15 yrs after final degree), who has personally conducted his or her own research, and who has made a significant contribution." This award will be awarded at the ASM/TMS Fall Meeting in Rosemont, Illinois, October 1998.

- Lumley Research Award for 1999 was awarded to M. J. Mills from the College of Engineering, The Ohio State University. This award is in recognition of outstanding achievement in research activities .

- M. J. Mills was awarded a 2001 Miller Visiting Professorship from the Miller Foundation of the University of California, Berkeley. Professor Mills spent a three-month period from July through September, 2001 at Berkeley working in conjunction with Professors Chrzan and Morris in the Department of Materials Science and Mineral Engineering.