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13. ABSTRACT *(Maximum 200 words)*

Superplasticity in crystalline solids is an area of expanding scientific and technical interest. Superplastic materials are characterized by a high value of the strain-rate-sensitivity exponent, m. Two kinds of superplasticity have been identified: fine structure superplasticity (FSS) and internal stress superplasticity (ISS). In the case of FSS materials, a strain-rate-sensitivity exponent equal to about 0.5 is usually found and these materials deform principally by a grain-boundary-sliding (g.b.s.) mechanism accommodated by slip, involving dislocation climb. It is shown, however, that FSS materials can be appropriately alloyed to achieve a value of m equal to one, i.e. ideal Newtonian-viscous flow. This occurs in fine grained Class I solid solution alloys where the deformation process is g.b.s. accommodated by solute-drag-controlled slip. The reason why Newtonian-viscous flow is achieved is because no pile-up stress is generated as in g.b.s. accommodated by dislocation climb. In the case of ISS materials, the strain-rate-sensitivity exponent is always unity, i.e. they exhibit Newtonian-viscous flow. ISS materials need not be fine-grained, and generally deform by a slip deformation mechanism. This type of superplasticity is observed in materials that develop internal stress under thermal cycling conditions. It is shown that a normally brittle-like Al-SiC_W composite can be made superplastic, exhibiting 1100% elongation, under thermal cycling conditions. A physically-sound theory has been developed which quantitatively predicts the creep behavior of ISS materials.

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NEWTONIAN VISCOUS FLOW AND SUPERPLASTICITY IN FINE GRAINED METALLIC ALLOYS (N-00014-82-K-0314)

FINAL REPORT (March 1, 1982 to October 31, 1990)

by

Oleg D. Sherby, Eric Taleff and Shih-Chung Cheng

I. INTRODUCTION AND SUMMARY

The program sponsored by the U.S. Office of Naval research has centered upon understanding the superplastic behavior of metal-base alloys. The work covered two types of superplasticity: fine structure superplasticity and internal stress superplasticity. A number of metal-base alloys have been studied including: (1) PM aluminum alloys, (2) metal-matrix composites based on Al-silicon carbide whiskers, (3) two phase Fe-Al-C alloys, (4) two phase Mg-9Li alloys (with and without B_4C particle additions), and (5) two phase titanium aluminides (TiAl and Ti_3Al).

The O.N.R. sponsored research has proved to be highly productive. A summary of the most important results is given under item II of this report. Forty-four publications have resulted from the research work performed on the program and these are listed under item III. The principal investigator received a number of awards during the tenure of this program and these honors are listed under item IV.

A number of graduate students participated in the program and contributed significantly to the research findings including the following (with degrees at the Master, Engineer and Ph.D. level). (a) Master degree: David Selby, Scott Daniels, Robert McCann, Pascal Metenier, Shih-Chung Cheng and Eric Taleff. (b) Engineer Degree: Oscar A. Ruano, Jose Belzunce and H. Fukuyo. (c) Ph.D. degree: M. Y. Wu, Glenn Daehn and H. C. Tsai. Post-doctoral fellows and visiting scholars contributing to the research effect were Drs. Jeffrey Wolfenstine, Oscar A. Ruano, Gaspar Gonzalez-Doncel, Soon Hong and Dong Shin. Dr. Jeffrey Wadsworth, (Consulting Professor in the Department of Materials Science and Engineering) interacted with a number of the participants and his help was much appreciated. In addition to the above individuals, the O.N.R. program supported several undergraduate students, a number of whom were minorities. All these students, mostly juniors and seniors, benefited from interactions with the graduate students and with the principal investigator. They were: Karl Reese, David Porter, Steve McGriff, Richard Bean, Louis Vu and Amol Dhoshi. A number of these individuals have gone on to graduate school elsewhere in the country, mostly into industrial engineering, business and medicine.

During the period of the O.N.R. sponsored research, three individuals from the O.N.R. Washington office were monitors of the program. Dr. Bruce MacDonald was the program monitor from 1982-1986, Dr. Ralph Judy was the program monitor from 1986-1987, and Dr. George Yoder was the program monitor from 1987-1990. The principal investigator, and all participants, give their sincere

appreciation to these three scientific officers. They contributed significantly towards optimizing the ongoing research and towards assisting technically the group's studies on superplastic behavior of metal-base materials.

Interactions during the course of this program with Messrs. A. P. Divecha and S. Karmarkar of the Naval Surface Warfare Center at Silver Springs, Maryland proved mutually beneficial. Their contributions centered on the superplastic behavior of metal-matrix composites based on aluminum containing silicon carbide whiskers.

II. SUMMARY OF THE MOST IMPORTANT RESULTS

In the first four years of the program, the work centered on an understanding of the superplastic behavior of aluminum-base alloys. The studies included two types of superplasticity: fine structure superplasticity and internal stress superplasticity. The effort on fine structure superplasticity was to develop superior superplastic aluminum base alloys through proper choice of alloy composition involving powder metallurgy and thermo-mechanical processing. The flow stress-strain rate response was investigated for those aluminum alloys which were successfully prepared to obtain an ultrafine grain size (i.e. 2 to 4 μm). Experimental results yielded superplastic behavior of an Al-5Mg-1.2Cr alloy at $10^{-2} s^{-1}$ with greater than 1000% elongation. This combination of high ductility at a significantly high strain rate was remarkable when reported in 1987. Mechanisms of plastic flow during superplastic deformation were established; the objective here was to develop realistic constitutive relations that will permit predicting the optimum conditions of structure, temperature and strain rate where superplasticity will be observed. In studies on internal stress superplasticity, the mechanism of ductility-enhancement was investigated in normally brittle metal-matrix composites. The ONR sponsored work led to the first successful demonstration that fiber reinforced composites (such as Al-20% SiC whisker fibers) can be made ideally superplastic, i.e. Newtonian viscous in nature, when deformed under thermal cycling conditions. A maximum elongation of 1100% was achieved in a 6061Al-20%SiC_w composite. A unique, and physically sound, theory was developed which quantitatively predicts the behavior of materials under conditions where internal stress superplasticity can be expected. Detailed descriptions of the above results are given in a number of published papers (references 11, 13, 18, 19, 20, 22, 26, 30 and 32 listed in the publications section).

In the latter part of the ONR program, emphasis was placed on development of superplasticity in other metal-base alloys by several processing methods and by several alloying combinations. The work centered on a magnesium-lithium alloy, on an iron alloy containing aluminum and carbon, and on a titanium-aluminum alloy. In all cases, a large amount of second phase was present in a given matrix phase, and the end objective was to achieve ultrafine-grained structures.

Studies on a Mg-9Li alloy involved developing an ultrafine-grained material by foil metallurgy processing. The initial objective was to prepare foils from an as-cast ingot. The steps included extensive cold rolling with intermediate annealing steps at low homologous temperature. Deformation bands developed during cold rolling whose spacing diminished with the amount of cold rolling. No recrystallization was observed after the cold rolling and annealing steps. Recrystallization takes place only during press-bonding of the foils at 200°C. The final grain size obtained parallel the band spacing-cold reduction curve. A fine grain size of 2μm was achieved with a cold reduction of 200 to 1. It was shown that such a material is superplastic at intermediate temperatures and has outstanding tensile ductility properties at room temperature. The details of this study are given in references 34, 35, 36, 38 of the publications section.

The superplastic flow behavior of an ultrahigh carbon steel containing 10% aluminum and 1.25% carbon was studied by H. Fukuyo as part of his engineer degree thesis. He observed a strain-rate sensitivity exponent, m, that was considerable higher than the normally observed value of 0.5 and plastic flow behavior approached that of a Newtonian-viscous solid, that is, m=1. These results lead Fukuyo, Tsai, Oyama and Sherby (hereafter quoted as Fukuyo *et al.*) to assess if similar trends were observed in other superplastic metal-base alloys. Indeed, it was discovered that, although m=0.5 was typically observed when deformation occurs at intermediate temperatures and fine grain sizes, a different observation was noted at high homologous temperatures. Thus, at high temperatures, where the activation energy for superplastic flow (Q_c) is about equal to that for lattice diffusion., The strain-rate-sensitivity exponent is either equal to 0.5 or to a value greater than 0.5. (Examples are given in reference 40 of the publications section). Fukuyo *et al.* developed a model to explain the different results for m obtained for different fine-grained alloys at high temperatures. The model is similar to the Ball-Hutchinson, Mukherjee, Langdon concept based on a grain boundary sliding process accommodated by slip. The slip accommodation process involves the sequential steps of glide and climb. When climb is the rate controlling step, the strain-rate-sensitivity exponent is 0.5 because of the pile-up stress at the head of the climbing dislocation (this, in fact, is the prediction of the Ball-Hutchinson, Mukherjee, Langdon relations). When glide is the rate-controlling step, however, Fukuyo *et al.* showed that the strain-rate-sensitivity exponent is equal to unity because there is no pile-up stress. Since glide and climb processes are sequential, the slower of the two processes is rate-controlling. This model predicts that fine-grained Class I solid solution alloys can exhibit a high value of m equal to unity since in these alloys the glide step (solute-drag-controlled dislocation creep accommodating grain-bounding-sliding) is often the slower process. On the other hand, fine-grained Class II solid solution alloys, in which dislocation climb is the rate-controlling step, should exhibit m values that are equal to 0.5. The predictions of Fukuyo *et al.* have been confirmed for a number of fine-grained solid solution alloy systems studied at high temperature. The quantitative model predicts that a low chemical diffusion coefficient (aluminum diffusion in the case of the iron-aluminum-carbon alloy), fine grains and high temperature favor Newtonian-viscous flow.

Work was initiated on a titanium-aluminum alloy in the composition range where two aluminide phases coexist (TiAl and Ti_3Al). Fine grain sizes can be really achieved in such a material by thermal mechanical processing since transformations in this material are similar to hypereutectoid steels in which we have had a great deal of experience. This work is under continuing investigation by Shih-Chung Cheng and Jeffrey Wolfenstine.

The major prerequisite for superplasticity is attainment of a high strain rate sensitivity exponent, m. Ideal Newtonian-viscous flow, where m=1, is a desirable objective and we have considered various mechanisms to achieve this objective including: (1) internal stress superplasticity, (2) grain boundary sliding accommodated by solute drag dislocation glide, (3) Harper-Dorn dislocation creep and (4) Nabarro-Herring diffusional creep. Mechanisms (1) and (2) have been mentioned earlier in this final report. Mechanisms (3) and (4) have been assessed by us in a number of publications. As a way of summarizing our studies on Harper-Dorn creep, it is believed unlikely that superplastic behavior can be achieved at reasonable, i.e. commercially useful, strain rates when this mechanism is rate-controlling. An internal stress model was developed by Wu and Sherby which unified Harper-Dorn creep and power law creep into one relation. Nabarro-Herring diffusional creep, although well respected as a theoretical model, does not appear to be confirmed by hard experimental evidence. Rather, it would appear that in those cases where diffusional creep was considered to be a rate-controlling mechanism, the data are better correlated when relations for grain boundary sliding or Harper-Dorn creep are utilized. References 2, 10, 12, 14, 17, 24, 26, 27, 33 and 41 in the publications section are papers that cover the Harper-Dorn and diffusional creep mechanisms.

A number of reviews on superplasticity were prepared by the principal investigator and his collaborators, Oscar Ruano and Jeffrey Wadsworth. These reviews are listed as references 1, 6, 7, 8, 9, 16, 23, 25, 28, 31, 39 and 40 in the publications section.

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IV AWARDS AND HONORS

The opportunity to do advanced research work at Stanford through sponsored research programs has lead to recognition of our studies. Much of this recognition is related to sponsorship of research on mechanical behavior of solids by the Materials Division of the U.S. Office of Naval Research. The following honors and awards were received by the principal investigator during the period of this ONR sponsored program.

1. In 1984, Oleg D. Sherby was one of two recipients to be honored the rank of AIME Fellow. He was presented the Fellow Award at the AIME Annual meeting on 25 February 1985 in New York City. He was cited "for his recognized contributions of fundamental relationships between creep behavior and basic diffusion theory and for developing a new class of superplastically deformed high carbon steels".

2. In 1985, Oleg D. Sherby was recipient of the ASM Gold Medal. This award is the highest annual award given by the American Society for Metals and was established "to recognize outstanding metallurgical knowledge and great versatility in the application of science to the metal industry, as well as exceptional ability in the diagnosis and the solution of diversified metallurgical problems". Dr. Sherby received

the ASM Gold Medal at the annual ASM Materials Meeting, Toronto, Canada, 12-17 October 1985. He was cited for " pioneering work on diffusion in controlling the strength of materials at elevated temperature and for innovative development of superplastic ultrahigh carbon steel".

3. In 1987, Oleg D. Sherby received the Charles S. Barrett Silver Medal Award. This award was presented by the Rocky Mountains Chapter of ASM on November 5, 1987. The award was for "outstanding contributions to the field of metallurgy".

4. In 1988, Professor Oleg D. Sherby was presented the Yukawa Memorial Lecture Silver Medal Award. The award was presented by the Japan Iron and Steel Institute at the 73rd annual meeting held in Chiba, Japan on April 1, 1988. Professor Sherby's talk was on "Advances in Superplasticity and in Superplastic Materials".

5. In 1988, Professor Oleg D. Sherby was honored with the Albert Easton White Distinguished Teacher Award. Sponsored by ASM International, the award was presented at the World Materials Congress, Chicago, in October 1988. The award was given in recognition of "unusually long and devoted service in the teaching of metallurgy, materials science and engineering, and, is characterized by ability to inspire and to impart enthusiasm to students, as well as by materials accomplishments".