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13. ABSTRACT (*Maximum 200 words*)

Ultrafine grained iron carbide material was developed by an atomized powder process, utilizing hipping, pressing and extrusion procedures. The material was made superplastic and behaved like other superplastic ceramics. This observation lead to the following conclusions.

Superplastic ceramics and metallic alloys exhibit different trends in tensile ductility in the range where the strain-rate-sensitivity exponent, m, is high ($m \geq 0.5$). The tensile ductility of superplastic metallic alloys (e.g. fine-grained zinc, aluminum, nickel and titanium alloys) is primarily a function of the strain-rate-sensitivity exponent. In contrast, the tensile ductility of superplastic ceramic materials (e.g. zirconia, alumina, zirconia–alumina composites and iron carbide) is not only a function of the strain-rate-sensitivity exponent, but also a function of the parameter $\dot{\epsilon}\exp(Q_c/RT)$ where $\dot{\epsilon}$ is the steady-state strain rate and Q_c is the activation energy for superplastic flow. Superplastic ceramic materials exhibit a large decrease in tensile elongation with an increase in $\dot{\epsilon}\exp(Q_c/RT)$. This trend in tensile elongation is explained based on a "fracture-mechanics" model. The model predicts that tensile ductility increases with a decrease in flow stress, a decrease in grain size and an increase in the parameter $(2\gamma_s - \gamma_{gb})$, where γ_s is the surface energy and γ_{gb} is the grain boundary energy. The difference in the tensile ductility behavior of superplastic ceramics and metallic alloys can be related to their different failure mechanisms. Superplastic ceramics deform without necking and fail by intergranular cracks that propagate perpendicular to the applied tensile axis. In contrast, superplastic metallic alloys commonly fail by intergranular and transgranular (shearing) mechanisms with associated void formation in the neck region.

14. SUBJECT TERMS

Superplasticity, iron carbide, ceramics, fine-grained materials, hipped materials, atomized powders, tensile elongations

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SUPERPLASTIC CERAMICS
(with emphasis on iron carbide)
(DAAL-03-88-K-0056)

FINAL REPORT (May 1, 1988 to April 30, 1991)

by

Oleg D. Sherby, Jeffrey Wolfenstine and Woo Jin Kim

This final report summarizes the work accomplished during the period May 1, 1988 to April 30, 1991 on a program on superplastic ceramics with special emphasis on developing processing methods for creating ultra-fine structures in iron carbide, quantifying its microstructure and assessing its superplastic characteristics. The program was sponsored and monitored by the Army Research Office under Contract DAAL-03-88-K-0056. During the course of the program, five semi-annual reports were prepared describing the details of the work accomplished and the projected future plans. The project monitors of the program were Drs. George Mayer, Iqbal Ahmad and Edward Chen. The authors would like to express their appreciation for the encouragement, cooperation and technical suggestions provided by the project monitors. In the latter stage of the program, Dr. Chen was especially helpful in discussions on the importance of grain boundary, interphase boundary and surface energy contributions to the enhancement of superplasticity in fine grained two phase ceramics.

1. Statement of the Problem Studied

The two principal objectives of this program are (1) to develop processing methods for achieving stable ultrafine grained (in the order of one micron) carbide-base materials, and (2) to evaluate and understand the mechanical behavior of ultrafine grained carbides at high temperatures, with special attention to superplastic flow and failure mechanisms.

2. Summary of the Most Important Results.

The initial objective of selecting a suitable composition, and appropriate processing procedures to achieve an ultrafine grained iron carbide base material was successful. A 2 to 3 micron grain size iron carbide matrix material containing about 20 percent of a discontinuous phase of iron was obtained. The second phase was considered essential to maintain a fine grain size at elevated temperature. An atomized powder approach was used, and consolidation was by hipping, compacting and extrusion procedures. The rapid-solidified powders and their extrusions were done in collaboration with Professor George Frommeyer of the Max Planck Institue fur Eisenforschung GMBH, Dusseldorf, Germany. The hipping procedures were done in collaboration with Dr. Oscar Ruano of the National Center for Metallurgical Research in Madrid, Spain. Both Frommeyer and Ruano spent a number of months at Stanford as visiting scholars (with their own funds) collaborating on the ARO program.

An important finding was that high strain-rate sensitivity was achieved in the iron-carbide material at elevated temperature, and tensile tests revealed that the material was superplastic. Elongations as high as 610% were achieved, and the grains were shown to be remarkably stable and equiaxed even after extensive deformation. It was concluded that the principal deformation mechanism was that of grain boundary sliding accommodated by slip processes near the grain boundary. This is the same mechanism that has been proposed by us in studies on superplastic metallic materials.

The most significant finding was that superplastic ceramics behave differently from superplastic metallic alloys with respect to tensile ductility. Most superplastic metallic alloys show high tensile elongations as long as the strain- rate- sensitivity exponent is high. This is not true with superplastic ceramics. It was shown by us that fine-grained superplastic ceramics exhibit tensile elongations that are a function of the stress even when the strain-rate-sensitivity exponent is high. Specifically, as the stress is increased the elongation to fracture decreases. We have developed a fracture mechanics model which predicts the fracture trend observed not only in the iron carbide material studied but in all superplastic ceramics studied to date. In the fracture mechanics model developed (Acta Metall. et Materialia, 1991) we show the tensile ductility increases as the flow stress is decreased, as the grain size is decreased and as the difference between the surface energy and the interphase boundary energy is increased. The quantitative relation developed yielded the following conclusion: if iron carbide can be processed to achieve an ultrafine stable grain size of 0.1 microns, then a tensile elongation of 8900 percent is achievable (this is the prediction for a strain rate of 5×10^{-4} s^{-1} and a temperature of 950 C).

The difference in the tensile ductility behavior of superplastic ceramics and metallic alloys can be related to their different failure mechanisms. In superplastic metallic alloys, failure is commonly associated with voids involving intergranular and transgranular mechanisms, in the neck region, that grow parallel to the tensile axis, while superplastic ceramics deform without necking and fail by intergranular cracks that propagate perpendicular to the applied tensile axis.

During the entire program, continuous and close interactions have occurred between the Stanford group and Drs. J. Wadsworth and T.G. Nieh of the Lockheed Palo Alto Research Laboratories. Several joint publications on superplastic ceramics have resulted as a consequence of this collaboration.

In the following section, we list the publications that have resulted from studies performed on this program, as well as from close interactions with our Lockheed collaborators.

3. List of publications.

(1) "Superplastic Behavior of Iron Carbide", by W.J. Kim, J. Wolfenstine, G. Frommeyer, O.A. Ruano and O.D. Sherby, Scripta Metallurgica, 23, 1989, 1515.

(2) "Superplasticity - Recent Advances and Future Directions", by Oleg D. Sherby and Jeffrey Wadsworth, Progress in Materials Science, 33, 1989, 169-221.

(3) "A Thermal-chemical Etching Technique for Hypereutectic Iron", by J. Wolfenstine, R.P Kershaw, W.J. Kim and O.D. Sherby, Materials Characterization, 24, 1990, 375.

(4) "Observations on Historical and Contemporary Developments in Superplasticity", by Oleg D. Sherby and Jeffrey Wadsworth, in Superplasticity in Metals, Ceramics and Intermetallics, M.J. Mayo, M. Kobayashi and J. Wadsworth, Eds., Materials Research Society, 196, 1990, 3-14.

(5) "Superplastic Properties of Hipped and Extruded Iron Carbide", by W.J. Kim, O.A. Ruano, J. Wolfenstine, G. Frommeyer and O.D. Sherby, in Superplasticity in Metals, Ceramics and Intermetallics, M.J. Mayo, M. Kobayashi and J. Wadsworth, Eds., Materials Research Society, 196, 1990, 359-364.

(6) "Superplastic Behavior in Ceramics, Ceramic Composites, Metal Matrix Composites, and Intermetallics" by T.G. Nieh, J. Wadsworth and O.D. Sherby, in Superplasticity in Aerospace II, T.R. McNelley and H.C. Heikkenen, Eds. The Minerals, Metals and Materials Society, TMS-AIME, Warrendale, Pa. 1990, 19-32.

(7) "Tensile Ductility of Superplastic Ceramics and Metallic Alloys, by W.J. Kim, J. Wolfenstine and O.D. Sherby, Acta Metallurgica et Materialia, 39, 1991, 199-208.

4. All participating scientific personnel.

Dr. Oleg D. Sherby, principal investigator

Dr. Jeffrey Wolfenstine, post-doctoral fellow

Mr. Woo Jin Kim, graduate research assistant

Dr. Jeffrey Wadsworth, Consulting Professor

Prof. Georg Frommeyer, Visiting Scholar

Dr. Oscar A. Ruano, Visiting Scholar

5. Theses.

Mr. Woo Jin Kim, Ph.D. to be completed by end of 1991.

Dr. Jeffrey Wolfenstine is now Assistant Professor in the Mechanical Engineering and Aerospace Department, University of California, Irvine, CA., 92717.