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ON DISPERSION STRENGTHENING OF ZIRCONIUM

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As a possible method of improving the elevated temperature strength properties of zirconium, an investigation was initiated on the feasibility of producing a dispersion-strengthened material. Because of the high reactivity of zirconium, its high solubility for oxygen, and the stability of the zirconium-oxygen solid solution, the major problem in such a development is choice of a suitable dispersant. That is, the dispersant must not react with the matrix at temperatures required for fabrication or at temperatures encountered during service. Thus, in the present investigation we were concerned primarily with the stability of various dispersants in the zirconium matrix.

The procedure employed in this study consisted essentially of blending powders of zirconium and a dispersant, followed by cold compaction and extrusion. The best available source of -325 mesh zirconium powder contained between 1500 and 3500 ppm O_2 ; to this material, powders of zirconium carbide (5μ max.), vanadium carbide (5μ max.), alumina (0.03μ average), lanthana ($0.8-2\mu$), ceria ($0.8-2\mu$), yttria ($0.8-2\mu$), calcia (44μ max.), or thoria ($0.6-0.8\mu$) were added in amounts of 7 vol % and compacted at 65 tsi. A cylindrical "green" compact having a density between 70 and 80 per cent of theoretical and dimensions of 1 inch diameter by 1 1/4 inch high was thus produced. After canning in copper, these billets could be extruded (5:1) on a 54-ton capacity, slow traverse hydraulic press with

the billet temperature at 1550°F and the extrusion die at 900°F. However, lack of densification of some of the extruded billets necessitated ratios of at least 23:1. For this high area reduction, the small size, slow press was not capable of extruding dispersed materials; only zirconium powder with no added particles was successfully extruded at high ratios using this press. This observation immediately indicated a pronounced strengthening effect due to the particles. Ultimately, all billets were successfully extruded at 1500°F to 0.190 inch diameter, fully dense rods (32:1) using a 200-ton capacity press and a ram speed of 90 ipm.

The microstructure of as-extruded Zr-7 vol % La_2O_3 and Zr-7 vol % Y_2O_3 are shown in Figures 1(a) and 1(c), respectively. These structures are similar to all the as-extruded materials and consist of an elongated, single-phase zirconium matrix dispersed with oxide or carbide particles.

Very little porosity is observed, and the interface between matrix and particles appears satisfactory. The size and distribution of particles, however, are not ideally of the type desired for strengthening by a dispersion of particles. Since the primary study was of particle stability with respect to zirconium, no attempt was made to overcome agglomeration during powder blending and fabrication. Some areas exhibit

Fig. 1 - Microstructures of particle-dispersed zirconium, as-extruded and after annealing. Unetched, X500. (a) Zr-7 vol % La_2O_3 as-extruded; (b) Zr-7 vol % La_2O_3 annealed 1600°F , 10 days; (c) Zr-7 vol % Y_2O_3 as-extruded; (d) Zr-7 vol % Y_2O_3 annealed 1600°F , 10 days. These oxide particles remained stable in the zirconium matrix.

fine, well-distributed particles; undoubtedly, a small proportion is submicroscopic and has the proper size (about 0.01μ) and particle spacing (0.01 to 1μ) for optimum strengthening.

The ambient temperature for service application of these materials is 1200°F . To test particle stability, specimens of as-extruded materials were annealed in sealed, helium-filled bulbs at 1600°F for 10 days. Any dispersants which show stability in zirconium at 1600°F would undoubtedly exhibit stability at the lower service temperature. The microstructure of the lanthana and yttria dispersed materials are shown in Figures 1(b) and 1(d), respectively, after annealing at 1600°F for 10 days. These dispersants remained stable in the zirconium matrix; there is no indication of particle reduction, agglomeration, rounding, or solution of these oxides in zirconium after the annealing treatments. Essentially, there is no difference in microstructure before or after annealing (except for matrix recrystallization, which is not shown). The specimen containing 7 vol % ThO_2 behaved in a similar manner; however, the other specimens containing oxides and the carbide-dispersed materials showed evidence of particle instability. Such behavior was manifested by change in the matrix area surrounding a particle, grain boundary penetration much like that observed for liquid-phase boundary

wetting, particle rounding and agglomeration, dissolution of the particles, or hardness increase.

Tensile and stress-rupture specimens having a gage diameter of 0.100 inch were machined from the as-extruded rods and evaluated at 1200°F. A summary of data on those materials considered promising from the standpoint of particle stability is presented in Table I. The value of tensile strength for undispersed zirconium is slightly higher than data reported elsewhere;^(1, 2) however, the latter data are not in agreement. While the elongated structure of extruded rods might account for the slightly high tensile strength at 1200°F, the high oxygen content (about 3000 ppm) probably does not cause strengthening at this temperature. According to Rubenstein,⁽³⁾ oxygen has only a very small effect on hardness of zirconium at 1100°F and therefore probably would not influence tensile and stress-rupture properties at 1200°F. The tensile strength and the 100-hour stress-rupture strength of dispersed materials are higher than the strength of undispersed zirconium, and this behavior is undoubtedly due to the particle additions. It must be emphasized that neither the ideal particle size nor optimum inter-particle spacing exists in the microstructure except for perhaps a very small percentage of particles. No attempt was made to optimize particle size or amount

TABLE I. Mechanical Properties of Experimental Zirconium Alloys
at 1200° F.

Composition, vol %	Ultimate Tensile Strength, psi	Yield Stress, psi	Elongation, %	100-hour Rupture Strength, psi
Undispersed Zr	8,600	8,400	37.5	2,000
Zr + 7 La ₂ O ₃	14,000	13,000	15	--*
Zr + 7 Y ₂ O ₃	14,600	14,600	36	2,800
Zr + 7 ThO ₂	16,200	16,200	23	3,600

* A threaded specimen was required for stress-rupture tests; it was not possible to machine threads on the lanthana-dispersed material.

of particles added. With the total amount of particles existing as an optimum or "critical dispersion," strengths of much higher magnitude would be anticipated. Since some particle strengthening was noted and since particle stability was demonstrated to a certain extent, it appears that dispersion strengthening of zirconium is feasible.

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