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Early work on rapidly solidified uranium

Rapid solidification is receiving considerable attention as a means of obtaining novel microstructures in metals and increasing the options available in alloy compositions [1]. Notably, rapid solidification can produce extremely fine grain structures and amorphous materials (glassy metals). The widely recognized value of rapid solidification prompts this summary of work carried out in the early 1960's on the application of this approach for the search for novel structures in uranium. The original report on this work [2] was classified and first became widely available in 1975.

This work was undertaken early in the development of the rotating electrode process (REP), which is now well established for the preparation of spherical powders of a wide range of metallic compositions [3, 4]. This method is based on the ejection of molten droplets from an electrode rotating rapidly while being arc-melted. Freezing of the droplets in flight yields spherical powder. From the outset, it was recognized that interception of a molten droplet by a solid surface effects more rapid dissipation of the heat of solidification. The flattened droplet can acquire a more refined structure as a result of the accelerated solidification. Applications of the process to other metals are cited in the annotated bibliography of Jones and Suryanarayana [5].

The effect of impingement of molten uranium droplets on solid copper was explored for its effect on the microstructure of the particulates collected and designated as "splat". The techniques developed were primarily concerned with (1) most effective cooling by the copper collector and (2) removal and recovery of individual splats from the collector so that succeeding droplets would impinge on a bare cold copper surface rather than build up on a preceding splat. Features incorporated to deal with these concerns included the following:

- (1) The $\frac{1}{2}$ -inch thick copper collector was cooled with helium pre-cooled by liquid nitrogen. This collector rotated at 1750 rpm in a direction to move the deposit away from the melting region of the rotating electrode.
- (2) Hollow unalloyed uranium electrodes (2-inch o.d. by 1 5/8-inch i.d.) were rotated at 7000 rpm. The current was turned on for 5 to 10 sec bursts, between which the collector was given the opportunity to recover its cooling capability and bare surface.

Optical metallography of splat prepared in this way showed it to have a mixture of very fine grains (below $10\,\mu\text{m}$) and a structure that was more difficult to resolve. It was then described as "acicular" and approximated what would now be called "microcrystalline". No attempt was made to estimate the cooling rate. Test samples were heated

in a hot-stage microscope for an hour at either 550 or 600° C. At either temperature, the acicular structure was transformed. Extremely fine grains were retained at 550° C but the 600° C treatment coarsened the grains to diameters of about $20 \,\mu\text{m}$.

Consolidation of splats to full-density rods was successfully achieved by cold compaction followed by extrusion (reduction of 8:1 or 23:1) at either 510 or 605° C.

Details regarding processing and evaluation are given in the report by Jordan [2]. In case of renewal of interest in rapidly-solidified uranium, further work would apply a more refined version of REP or other means of rapidly quenching metals. Nonetheless, the early work by Kaufmann, Jordan and their colleagues at Nuclear Metals, Inc., is worth recording as a useful point of departure.

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Room-temperature fracture energy of monoclinic Gd_2O_3

Fracture energy data for non-cubic polycrystalline materials is relatively limited. Graphite and $Al_2\,O_3$ have been studied extensively, but the bulk of the fracture energy work has been carried out on cubic systems. For the rare-earth oxides in particular, no fracture energy data is available on either the cubic or non-cubic oxides. In this study the fracture energy, γ_f , was measured as a function of porosity for monoclinic $Gd_2\,O_3$.

In order to prepare the Gd₂O₃ specimens, gadolina powder from the Ames Laboratory Rare Earth Separation Group* was dissolved in nitric acid and precipitated according to a technique developed by Dole et al. [1]. The powder was then hot-ground in a mortar and pestle and calcined at 1000° C in an alumina crucible. The powder was dried for 2 h at 150° C and then pressed at 3.45 MPa (500 psi) in a double-action steel die having a rectangular cavity of 7.6 cm × 0.6 cm. Following dry pressing, the resulting bars

were enclosed in rubber containers and isostatically pressed at 206.8 MPa (30 000 psi).

The $\mathrm{Gd}_2\mathrm{O}_3$ specimens were sintered in air in a gas-fired furnace at 1300 to 1650° C for 1 to 6 h. In order to limit specimen contamination, the bars were placed on a 2 mm thick bed of $\mathrm{Gd}_2\mathrm{O}_3$ powder inside a horizontal muffle tube. Prior to sintering, the specimens were covered with an additional 2 mm layer of $\mathrm{Gd}_2\mathrm{O}_3$ powder.

The fired specimens were diamond ground into rectangular prisms having a dimensional tolerance of ± 0.005 cm. The resulting bars, which were about 5.0 cm long, 1.0 cm in depth and 0.5 cm thick, were then cut lengthwise producing two bars of nearly identical microstructure, each about 0.2 cm thick after regrinding. The specimens were then annealed in air at 1000° C for at least 8 h.

The room-temperature Young's modulus was measured for each specimen using the sonic resonance technique originated by Forster [2]. The experimental technique has been discussed in detail by Spinner and Teft [3] and a description of the apparatus is available elsewhere [4, 5]. The

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