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Forming rapidly cooled materials using a laser beam

The formation of metastable alloy phases by rapid cooling was demonstrated by Duwez *et al.* [1, 2] in an apparatus in which a molten globule was ejected from a melting chamber onto a rotating cylinder or a stationary copper target. Similar rapidly cooled alloys of more uniform thickness were made by Pietrokowsky [3] employing an anvil device in which the samples were melted by a gas flame. Laser usage as a source of heat has also produced rapid quenching of metallic alloys [4–7]. A complex triggered device was reported by Krepski *et al.* [8] which incorporated both the laser and a triggered hammer-anvil technique for quenching of their specimens.

This paper describes experiments in a simplified apparatus which employs a laser beam for heating and melting a specimen and copper-cooled anvils for the then rapid cooling of the molten globule (Fig. 1).

The advantages of this device are: (1) high meltting point materials can be melted with a reasonable power laser, (2) the oxidation of the specimen is minimized because the entire operation is extremely rapid, (3) there is no container problem since the specimen rests on a relatively massive copper plate, (4) no complex triggering device is needed to close the anvils because the laser beam can be kept on while the copper anvil is in motion.

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The sample is placed on a copper plate which serves as a broad track for the moving anvil. The specimen is melted with the prefocused laser beam and the solenoid is then activated. The moving anvil sweeps the molten specimen along the track and against the stationary cooled specimen plate forming a thin foil.

Melting of samples was accomplished using a continuous, convectively cooled, high-power CO_2 laser of 10.6 μ m output wavelength. The unit used was a 12 tube laser built by United Technologies Research Center which could be operated in any one of three output modes. As an oscillator/ amplifier (Gaussian output energy distribution), the unit is nominally specified as a 6 kW laser. As an unstable resonator (hollow output beam), the laser has produced 9 kW and in the "top hat" mode (uniform energy distribution) the laser has produced powers to 10 kW. Power densities by any of



Figure 1 Simplified apparatus for forming rapidly cooled material.

MOHAMMAD A. SALAMAH D. WHITE Department of Ceramics, The University, Leeds, UK these energy distributions at the focal point even at 3 kW total output power have been sufficient to melt the small material quantities used to form the foils. Focusing was typically accomplished with a concave mirror having an 18 in. focal length. Melting is rapid, with minimal contamination and insufficient time for heating of the copper substrate. Splatting was done only 1 to 3 sec following onset of melting, and reaction of the splat charge with the copper substrate was found to be essentially nonexistent. A blanket of inert gas can be provided for highly reactive alloys. In order to provide finer melting control, 3 kW was used for splatting in all tests.

A large number of alloy compositions including the alloys of Ti-Ni, and Pd-Cu-Si discussed here have been successfully laser-melted and splatquenched. The convenience in making thin foils from splats has facilitated their examination by transmission electron microscopy. A macrograph of a typical laser-melted, splat-quenched specimen is shown in Fig. 2. The predominant effects of splat-quenching were to greatly refine the microstructures of the as-cast alloys, and to produce improved homogeneity of phases on a fine scale. Two examples of improved homogeneity are shown in Figs. 3 and 4.

In Fig. 3, the initial, as-cast alloy of 70 wt %



Figure 2 Typical quenched specimen, \times 4.5.

Ti-30 wt% Ni exhibits a structure consisting of primary α Ti dendrites surrounded by a eutectic consisting of alternate lamellae of α and a Ti-Ni intermetallic compound which is most probably Ti₂Ni. The α -phase contains some Ni in solid solution, having transformed from the β Ti-Ni solid solution in the solid state. After laser splat quenching, the resultant alloy exhibited considerable refinement, with an average grain size of <0.1 μ m. Phase homogenization also was noted, with the splat-quenched alloy consisting of a single phase – a supersaturated solid solution of Ni in Ti.

Shown in Fig. 4 is a thin foil and electron dif-



Figure 3 Refined grain structure, 70 wt % Ti, 30 wt % Ni alloy.



Figure 4 Amorphous thin foil, 90.65 wt % Pd, 4.22% Cu and 5.13 wt % Si alloy.

fraction pattern from a splat-quenched sample of an alloy consisting of 90.65 wt % Pd, 4.22 wt % Cu, and 5.13 wt % Si. The absence of structure within the foil and the diffused nature of the diffraction pattern indicate that a completely amorphous phase was formed as a result of the rapid cooling experienced during splat quenching.

Fractographic evidence of mixed mode stress corrosion cracking in stainless steel surgical implants

AISI stainless steel 316L is the most widely used alloy for orthopaedic surgical implants and in the majority of the cases the implants are used to hold bone sections while the bone structure reforms. The implants are subjected to significant stresses and are exposed to aqueous body fluids containing about 0.1 M chloride ion. The pH of the body fluid is normally 7.4 but under certain conditions a local variation in the range of 4 to 9 may result.

A significant number of implants fracture in service and the failure is found to occur mostly in plane strain. Upon examination of failed implants, a number of investigators [1-5] identified fatigue and metal defects as the cause of failure but evidence of pitting corrosion [6], crevice corrosion [7, 8] and intergranular corrosion [8] have also been observed on fracture surfaces. A problem faced by the investigators is that the features of

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> R. D. VELTRI E. M. BREINAN G. P. McCARTHY F. S. GALASSO United Technologies Research Center, East Hartford, Connecticut 06108, USA

interest on the fracture surfaces are frequently obliterated by the repeated impingements of the surfaces during the period after fracture before removal.

Recently, we received a number of failed stainless steel implants (mainly hip prostheses) and in two of these the fracture surfaces appear to be almost intact. Evidence of fatigue and pitting corrosion can be seen on the fracture surfaces of the implants and, moreover, there is evidence of what appears to be stress corrosion cracking (SCC) – both intergranular and transgranular crack propagation. This communication presents fractographic evidence of the mixed mode stress corrosion cracking. Although cases of mixed mode failure in SCC of austenitic stainless steel are reported in the literature, they are mainly limited to failures at the elevated temperatures whereas in the present case, failure took place at a rather low temperature, i.e. body temperature (37° C) . It is believed that the fractographs presented are of interest from this point of view.