ELECTROSLAG REFINING AS A CLEAN LIQUID METAL SOURCE FOR ATOMIZATION AND SPRAY FORMING OF SUPERALLOYS

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Abstract

A pilot plant has been constructed to demonstrate the concept of using a combination of electroslag refining (ESR) and an induction-heated, segmented, water-cooled copper guide tube (CIG) to melt, refine, and deliver a stream of liquid metal to a spray forming process. The basic ESR system consists of a consumable electrode of the alloy to be melted, a liquid slag, and a water-cooled copper crucible. The liquid slag is heated by passing an ac-electric current from the electrode through the slag to the crucible. The liquid slag is maintained at a temperature high enough to melt the end of the electrode. As the electrode melts, a refining action takes place—oxide inclusions are exposed to the slag and are dissolved. Droplets of molten metal fall through the slag and are collected in a liquid metal pool contained in the crucible below. By the addition of the induction-heated, segmented, water-cooled copper guide tube (CIG) to the bottom of the crucible, a liquid metal stream can be extracted from the liquid metal pool. This stream makes an ideal liquid metal source for atomization and spray forming. The pilot plant has been operated at a melt rate of 15 to 25 kg/min with the Ni-base superalloys Alloy 718, Rene' 95 and Rene' 88. Process optimization and cleanliness evaluation studies are in progress.

Introduction

Spray Forming

Spray forming is being considered as a low-cost alternative to *powder metallurgy* for the preparation of high-strength forged superalloy components. Spray forming is the term applied to a process in which a stream of liquid metal is gas-atomized and then immediately reconsolidated into a solid shape [1, 2]. In this process, the atomized droplets of liquid metal are cooled by the atomizing gas, so that they lose most (75%) of their heat of fusion while in flight from the atomizer. Upon striking the solid shape, the metal quickly loses the balance of its heat of fusion such that only a thin layer of liquid metal is maintained on the surface throughout the process. The spray forming process has been studied extensively for nickel-base superalloys [3, 4], and has been shown to be capable of producing a homogeneous fine-equiaxed-grain microstructure (30- to 40- μ m grain diameter), with a relative density of greater than 98%.

Ceramic Inclusions - A Problem

Ceramic inclusions can reduce low-cycle-fatigue (LCF) life in both the *powder metallurgy* approach and the *spray forming* approach to the preparation of superalloys [5, 6]. Mechanistically, a large ceramic inclusion will crack early in life and act as a crack starter for the surrounding superalloy. Direct observation of this event is shown in Figure 1. The aluminum oxide particle cracked very early in life and the crack rapidly extended into the surrounding superalloy.

Figure 1. Polished surface view of an aluminum oxide particle acting as a crack starter in the superalloy Rene' 95 mechanically loaded under low-cycle-fatigue conditions. [7]

Applying the discipline of fracture mechanics to this case, it can be shown that the higher the mechanical loading during the fatigue exposure, the smaller the cracked oxide must be to achieve equivalent life. The results of such a computation are shown in Figure 2.

The challenge from a processing point of view, therefore, is to limit the size of the largest ceramic inclusion that reaches the

Figure 2. Stress required to propagate a crack from an initial defect size to the critical crack size in 104 cycles. da/dn and K_{IC} taken as the average of values for Rene' 95, IN 100, Astroloy, Waspaloy, and INCO 901 at 650°C. [8]

final component. In the case of the powder metallurgy approach, this challenge is met by *sieving* the powder after *vacuum induction melting* and *gas atomization*; by careful handling during *canning* to place the powder in a container suitable for reconsolidation; by *vacuum degassing and sealing* to remove and prevent re-entry of the ambient atmosphere from the can; and by *extrusion* to reconsolidate the alloy to a fine grain billet [5].

ESR - A Solution

In the case of the spray forming approach, a novel melting system is being developed to make use of electroslag refining (ESR) to dissolve ceramic inclusions before the liquid metal is converted to droplets by gas atomization [9-15]. A 15-kg/min pilot scale system has been constructed and is being used to produce material for testing and evaluation [16-19].

Pilot Scale System

Concept

The pilot scale system being used to evaluate this approach is shown schematically in Figure 3.

Figure 3. Schematic diagram showing the general arrangement of the major components of the ESR-CIG pilot plant configured to deliver liquid metal to the atomizer of a spray deposition system.

The basic concept makes use of electroslag refining (ESR) to melt and remove ceramic inclusions from the superalloy, combined with an induction-heated, segmented, water-cooled copper guide tube (CIG) to deliver a stream of liquid metal to a spray forming atomizer. ESR removes ceramic inclusions in the following manner—liquid slag (calcium fluoride, calcium oxide and aluminum oxide) is heated by passing an ac-electric current from a consumable superalloy electrode (prepared by vacuum induction melting), through the slag to a water-cooled crucible. The liquid slag is maintained at a temperature high enough to melt the end of the electrode. As the electrode melts, a refining action takes place—ceramic inclusions are exposed to the slag and are dissolved. Droplets of molten metal fall through the slag and are collected in a liquid metal pool contained in the crucible below. The CIG system then delivers a stream of liquid metal from the crucible to the spray forming atomizer without reintroducing inclusions.

ESR System

The pilot scale system is equipped with an ESR unit that is a shortened version of a conventional ESR furnace [20]. The consumable electrode is 355 mm in diameter $\times$ 1800 mm in length and weighs approximately 1140 kg. The major components of the system consist of an electrode drive mechanism, a 430-mm-diameter water-cooled copper melt crucible that is only 600 mm tall (rather than 3000 to 5000 mm for a conventional system), a coaxial current return path (to minimize electromagnetic disturbance of the liquid metal stream), a 20-kA 1.6-kVA-power supply, and a melt-rate controller. The melt-rate controller uses a video imaging system to sense the metal-plus-slag height in the crucible and to maintain this level constant. A constant height is required to maintain a constant liquid-metal flow rate.

CIG System

The transfer of liquid metal from the ESR crucible to the spray forming atomizer is accomplished by an induction-heated, segmented, water-cooled copper guide tube (CIG). This type of guide tube was first developed for the transfer of liquid Ti-base alloys from a plasma-arc melting furnace (PAM) to the atomizer of a powder production system [21, 22]. In the current case, the induction heating is provided by a two-coil system, as shown schematically in Figure 4. A “starter plate” of previously refined material is placed at the bottom of the crucible, above the CIG, to isolate slag from the CIG. A hole is melted through the starter plate during the ESR/CIG start procedure.

The upper coil is driven by a 240-kW medium-frequency power supply (approximately 10 kHz). Its main function is to prevent re-solidification of liquid metal that fills the guide tube as the ESR starter plate melts. The lower coil is driven by a 110-kW high-frequency power supply (100 to 200 kHz) and is used to initiate flow of liquid metal by melting out a plug in the lower portion of the CIG once the correct metal head height is established in the ESR crucible, and to maintain superheat in the

Figure 4. Schematic diagram showing the general arrangement of the major components of the CIG system used to transfer liquid metal from the ESR crucible to the atomizer of a spray deposition system.

stream during the balance of the run. The liquid metal flow can also be turned off and on by varying the power level in the lower coil. The water-cooled guide tube is segmented into eight individual segments to allow the induction heating to reach the liquid metal inside. The segments are insulated from each other over most of their length, but are joined electrically at the outside diameter. Each segment is independently water-cooled.

Spray Deposition System

The spray deposition system includes a scanning atomizer, a preform handler, and a water-cooled deposition chamber. The scanning atomizer is a gas atomizer (nitrogen or argon) capable of converting the liquid metal stream into a spray of liquid metal droplets and directing this spray over a range of angles. The preform handler is a four-axis robot capable of positioning and rotating the preform beneath the spray. The preform may be held at any orientation relative to the atomized spray, allowing a high degree of flexibility in the angle of attack between the spray and the preform.

Preliminary Results

Liquid Metal Stream

The pilot scale system is operational. Figure 5 shows a liquid metal stream pouring from a 5-mm-diameter orifice at a rate of approximately 15 kg/min.

Figure 5. Liquid metal stream pouring from the induction-heated, water-cooled copper guide tube (CIG). The guide tube orifice is 5 mm in diameter. The liquid metal pour rate is approximately 15 kg/min.

A wide range of liquid metal flow rates can be achieved with this system. From energy balance considerations the mass flow rate of liquid metal exiting the CIG system, $\dot{m}$, can be expressed as

$$\dot{m} = A \rho_m C_D \sqrt{\frac{2 \delta p}{\rho_m} + 2g \left(h_m + \frac{\rho_s}{\rho_m} h_s \right)} \quad (1)$$

where A is the cross-sectional area of the outlet orifice of the CIG system, ρ_m and ρ_s are the densities of the liquid metal and the slag, δp is the difference in pressure between a point directly above the slag and a point directly below the outlet orifice, h_m and h_s are the corresponding heights of metal and slag, g is the acceleration caused by gravity, and C_D is an empirically determined drag coefficient. Measurements conducted with a ceramic-guide-tube system, under conditions where δp was controlled to maintain a constant equivalent metal head height [23], give a value of 0.84 for C_D . Liquid metal flow rates calculated using Equation 1, and the original data for the ceramic-guide-tube system are given in Figure 6.

Flow rates observed for the ESR-CIG system seem to follow this relationship quite closely.

Figure 6. Liquid metal flow rate as a function of guide-tube orifice area and liquid metal head height calculated using Equation 1. Data from the ceramic-guide-tube system used to determine the drag coefficient are also plotted [23].

Solidified Melt

A cross section of the solidified melt remaining in the crucible and the CIG after a run is shown in Figure 7.

Clearly visible in this photograph is the starter plate, with its corresponding heat-affected zone; the refined superalloy material, which fills the crucible and flows through the starter plate to fill the CIG; and the large-grain dendritic growth of the solidified melt. Some remnants of the unmelted slag are visible at the periphery of the solidified melt directly above the starter plate.

Cleanliness Evaluation

The electron-beam button test method [24] has been used to evaluate the capability of the ESR-CIG process to remove oxide nonmetallic inclusions from the superalloy Alloy 718. Electrodes were prepared as feedstock for this evaluation by vacuum induction melting (VIM). The specific oxide area (SOA) determined for these electrodes varied from 0.158 to 0.497 mm²/kg. This high level of SOA was reduced to 0.007 mm²/kg by passing the metal through the ESR-CIG process and solidifying it in an ingot mold. This low level is excellent and compares very favorably to the nominal level of 0 to 0.0488 mm²/kg normally observed for Alloy 718 prepared commercially by the "triple melt" process (VIM + ESR + VAR). Although SOA gives a relatively good measure of the total oxide volume, it does not give a good measure of the largest inclusion size to be found (the value most important for determining fatigue life).

Figure 7. Solidified melt remaining in the crucible and the CIG after the run.

Spray Forming

Initial spray forming trials have produced preforms in the 250 to 325 mm diameter range. One such preform is shown in Figure 8.

Figure 8. Spray formed preform. The dimensions are 300 mm in diameter $\times$ approximately 500 mm long. The weight is 270 kg. The alloy is Rene' 95.

The maximum length produced to date is 550 mm. The nominal yield (metal atomized divided by preform weight) is running in the range of 60 to 80%, with an average of 70%. The microstructures are equiaxed with an average grain size of 40 to 50 μm (with some

larger grains observed at the center location of some preforms). High levels of porosity are observed in the outer few millimeters of most preforms.

Process studies are in progress to optimize cleanliness, microstructure and yield. Electron-beam button tests are being used to measure the average level of cleanliness. Large-bar low-cycle fatigue tests are being used to determine the size of the largest inclusion that is able to survive the process.

Summary and Conclusions

A pilot plant has been constructed to demonstrate the concept of using a combination of electroslag refining (ESR) and an induction-heated, segmented, water-cooled copper guide tube (CIG) to melt, refine, and deliver a stream of liquid metal to a spray forming process. The basic ESR system consists of a consumable electrode of the alloy to be melted, a liquid slag, and a water-cooled copper crucible. The liquid slag is heated by passing an ac-electric current from the electrode through the slag to the crucible. The liquid slag is maintained at a temperature high enough to melt the end of the electrode. As the electrode melts, a refining action takes place—oxide inclusions are exposed to the slag and are dissolved. Droplets of molten metal fall through the slag and are collected in a liquid metal pool contained in the crucible below. By the addition of the induction-heated, segmented, water-cooled copper guide tube (CIG) to the bottom of the crucible, a liquid metal stream can be extracted from the liquid metal pool. This stream makes an ideal liquid metal source for atomization and spray forming. The pilot plant has been

operated at a melt rate of 15 to 25 kg/min with the Ni-base superalloys Alloy 718, Rene' 95, and Rene' 88. Process optimization and cleanliness evaluation studies are in progress.

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