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Characterisation of a complex thin walled structure fabricated by selective laser melting using a ferritic oxide dispersion strengthened steel

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ABSTRACT

Oxide dispersion strengthened (ODS) alloys exhibit superior mechanical and physical properties due to the presence of nanoscopic Y(Al, Ti) oxide precipitates, but their manufacturing process is complex. The present study is aimed at further investigation of the application of an alternative, Additive Manufacturing (AM) technique, Selective Laser Melting (SLM), to the production of consolidated ODS alloy components. Mechanically alloyed PM2000 (ODS-FeCrAl) powders have been consolidated and a fine dispersion of Y-containing precipitates were observed in an as built thin-walled component, but these particles were typically poly-crystalline and contained a variety of elements including O, Al, Ti, Cr and Fe. Application of post-build heat treatments resulted in the modification of particle structures and compositions; in the annealed condition most precipitates were transformed to single crystal yttrium aluminium oxides. During the annealing treatment, precipitate distributions homogenised and localised variations in number density were diminished. The resulting volume fractions of those precipitates were 25–40% lower than have been reported in conventionally processed PM2000, which was attributed to Y-rich slag-like surface features and inclusions formed during SLM.

1. Introduction

Modern ODS alloys have many potential fields of application. However, they were designed for high temperature applications, for instance in conventional power plants or in gas turbines, where an increase in operating temperatures is beneficial [1]. The portfolio of suitable materials decreases rapidly with increase of working temperature. Superalloys are capable of operating at temperatures of ~1000 °C [2], but this limit can be extended to up to 1300 °C for alloys containing an oxide dispersion [3]. Even higher operation temperatures are possible with ceramics, graphite, refractory metals and advanced thermal barrier coatings, but often their practical use is constrained [4,5].

ODS alloys have been developed over recent decades following the early work by Benjamin [6] on mechanical alloying (MA) of Ni-base alloys and are fascinating due to their interesting properties. Characteristic of this class of alloys is the presence of a fine dispersion of rare earth metal (typically yttrium) oxide precipitates having diameters of several nanometres. The current manufacturing route for ODS alloys includes MA of master alloys or elemental powder, hot extrusion or hot isostatic pressing (HIP), which can be followed by rolling and a final heat treatment [7]. Yttrium, which has a very low solubility in alpha-ferrite matrix at room temperature, is typically added to the alloy in the form of yttria (Y_2O_3) during MA. There is still some debate whether the yttria is broken down and Y is forced into supersaturated solid solution [8] or whether yttria just fractures and forms small amorphous compounds [9]. During successive consolidation, precipitates evolve in the alloy matrix and grow in diameter during various production steps [10] and amorphous compounds become crystalline [9]. The complex metallurgy route used to produce ODS alloys leads to high prices and limitations in the form of consolidated material, which motivates constant research on a number of alternative production routes, such as gas atomization reaction synthesis [11] or spark plasma sintering [12]. The excellent high-temperature oxidation resistance displayed by ODS-FeCrAl PM2000 alloy, as used in this study, has been attributed to the presence of yttrium and aluminium [13]. The fine precipitate dispersion blocks dislocation motion and the recrystallised microstructure, which is formed due to abnormal grain growth, is extremely coarse [14] and beneficial to creep resistance. The special resistance of ODS alloys to radiation induced damage has been investigated by ion implantation studies and evidence suggests that the ultrafine dispersoids can act as sink sites for transmutation He, formed when exposed to neutron irradiation [15]. For nuclear applications, however, the element Al needs to be minimised in order to avoid the formation of a long-lasting

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radioactive isotope and precipitates need to be in the nanometre size range [15]. Thus, ODS steels are considered as candidate structural materials for both fossil and nuclear energy applications. PM2000 was designed for high-temperature applications [16], but its use for biomass combustion [17] and medical implants [18] has also been considered.

Methods of joining of ODS alloys involving melting, usually result in agglomeration of the dispersoids and in inferior mechanical properties of the bond zone [19]; however, several solid-state joining techniques are currently being investigated with promising results [20]. An alternative method is laser beam welding and it was shown in several studies that ODS precipitates can be retained in the fusion zone [19,21]. This work uses SLM to consolidate as-MA PM2000 powder, which has certain similarities with laser welding. The technique produces fully dense solid freeform components by successive melting of thin layers, of metal powder (in this work: 50 μm), with a finely focused laser beam. The thermal conditions are similar to conduction based laser welding with typically very high cooling rates and continuous stirring in the melt pool caused by Marangoni-forces [22] making SLM of potential use in the ODS alloy environment. More details on the SLM process can be found elsewhere [23]. It was shown recently by Walker et al. that SLM builds grown using as-MA PM2000 powder contain a fine dispersion of precipitates in the alloy matrix and processing parameters can be altered to manipulate the particle size and minimise their degree of agglomeration [24]. Successive work in this field concentrated on ODS alloy coatings, which were well adherent to a substrate even when annealed [25,26] and the presence of fine Al and Y-rich precipitates in the deposit could be confirmed [27]. SLM processing parameters were further optimised to manufacture walls with different thicknesses and solid structures; and it was recently found that after a post-build anneal at 1200 °C for 1 h, a room temperature strength, which was comparable to that of conventional recrystallized PM2000, could be achieved [23]. However, the strength was inferior for Fe-base ODS-MA956 produced via a similar process, selective laser sintering (SLS) [28]. Processing parameters developed by Walker et al. [24] were used to fabricate the build in this study which is focused on more detailed characterisation since a profound understanding of SLM-ODS alloy fundamentals is still lacking. This study is part of a larger project investigating the feasibility of using SLM for additive manufacture of near net shape components, by consolidating mechanically alloyed powders from ODS alloys.

2. Materials and methods

2.1. Manufacture of SLM builds

The powder material consolidated in this work was supplied by Plansee GmbH (Reutte, Austria) in the form of as-MA ODS-PM2000. This material is no longer commercially available, but prior to use, the powder material was stored in sealed containers in dry conditions and chemical analysis did not reveal any form of contamination. The nominal composition according to the PM2000 datasheet [16] and measurements using inductively coupled plasma optical emission spectrometry (ICP-OES) are displayed in Table 1. In addition to the intended alloying elements, low levels of trace elements were also detected; for example, Mn and Ni were measured at 0.05 and 0.02 wt.% respectively. Other elements were measured at concentrations ≤0.01 wt.%, although oxygen levels could not be measured by this technique.

The same batch of PM2000 powder, used in this study, has been analysed in greater detail by Walker et al. [24]. The filling factor of the sieved powder was determined as 0.46, which means that in order to fabricate a consolidated layer of 50 μm, a 109 μm thick slice of powder needs to be scanned. Consequently, before the SLM procedure, the powder material was sieved with a 106 μm mesh size stainless steel sieve, in order to remove coarser powder particles. Consolidation was conducted on a SLM machine, type Realizer 100 from ReaLizer GmbH, which was equipped with a YLM-50 ytterbium fibre laser (LPG Photonics) and the maximum laser output power (50 W) was applied. The wavelength was 1075–1095 nm and the beam shape was similar to a Gaussian distribution. The beam was focused at the build area and the spot diameter was ~80 μm. The machine set-up, consisted of a sealed building chamber in which the consolidation process took place under Ar atmosphere with a remaining O concentration of 200–300 ppm prior to building. Oxidation effects with the alloy during processing resulted in a significant reduction of oxygen after the first few layers had been deposited. The inert gas was pumped through a filter system during processing and was re-directed through nozzles onto the build space. Scanning was conducted in directions toward the gas stream to minimise redeposition of ejected material which can affect the consolidation process. Parameters similar to those reported by Walker et al. [24], which produced the fewest oxide agglomerations in the build, were used in this study. The current scan strategy applied four parallel scans per deposit layer to form the wall thickness in order to obtain fully dense structures, but also to investigate the tendency for dispersoid agglomeration during re-melting sequences during growth. For each slice, the two outer lines were scanned first, followed by the inner two, in order to support the formation of dense walls, since powder particles are known to be dragged into the melt pool [29]. The distance between one outer and the neighbouring inner scan line was 70 μm, while the distance between the two inner lines was 60 μm. As presented in Fig. 1, the resulting build was a complex hexagonal shaped wall structure, which was grown on a mild steel substrate on triangular support structures, which eased the removal process.

2.2. Sample preparation and analytical techniques

In this study samples, taken from the outer walls, were sectioned using a water-cooled low speed saw. Specimens were chemically cleaned prior to heat treatments, which were conducted in a horizontal tube furnace on silica trays. Although ODS-FeCrAl alloys are known to form protective, slow growing, scales composed of α-Al_2O_3 at high temperatures (>~1000 °C [30]), a flowing argon atmosphere was used to protect against excessive oxidation during heat treatment.

Specimens were prepared for SEM analysis by standard techniques and a final polish was applied using a 40 nm colloidal silica suspension. Specimens which required etching were polished to a 0.25 μm finish, using diamond paste, prior to immersion in a 10% HCl 90% methanol solution. SEM work was conducted on a JEOL JSM-7001F field emission gun (FEG) instrument operated at beam energies of 15–30 keV in both secondary electron (SE) and backscattered electron (BSE) modes, respectively. Chemical analyses were performed by energy dispersive X-ray spectroscopy (EDS) on this machine using an INCA x-act-51-ADD0001 EDS system from Oxford Instruments.

Sections of the outer walls were thinned from both sides to a thickness between 100 and 200 μm. Discs 3 mm in diameter were punched and then ground, applying a 4000 grit finish, to a thickness of <50 μm. Final thinning to electron transparency was performed on a twin-jet electropolishing system, Tenupol-3 from Struers, in a 10% perchloric acid in methanol solution at a temperature of −45 ± 5 °C. Thin-foil samples from the top layers of the build could not be prepared by this method and were therefore prepared by a lift out method using a FEI Helios Nanolab 600i focused ion beam (FIB) instrument. Dispersoids were extracted onto amorphous C-film after a replica method described elsewhere [31] using a 10 vol.% HCl in methanol solution as an etchant. TEM imaging and EDS was conducted on a JEOL JEM-3010 instrument

Table 1
The elemental composition (wt.%) of mechanically alloyed PM2000 according to the alloy datasheet [16] (A) and according to an analysis of the powder by ICP-OES (B).

	Fe	Al	Ti	Cr	Y_2O_3	Y	O	Others
A	Balance	5.5	0.5	19.0	0.5	–	–	–
B	Balance	5.34	0.45	18.77	–	0.39	0.24	<0.15