Materials Characterization 145 (2018) 10–19

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

In-situ microstructural observation of Ti-Cu alloys for semi-solid processing

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ARTICLE INFO

Keywords:
Semi-solid processing
Thixoforming
Titanium alloys
HT-LSCM
Microstructure

ABSTRACT

The semi-solid processing of metallic alloys strongly depends on the microstructural features exhibited by the raw material. Consequently, many characterization techniques have been used to elucidate semi-solid microstructures. In the present work, high-temperature laser-scanning confocal microscopy (HT-LSCM) was used to characterize Ti-Cu alloys intended for thixoforming. During the reheating stage, this in-situ technique enabled us to observe the Ti-β grains formed through the reverse eutectoid transformation. Furthermore, the results showed that thermomechanical treatment was primarily responsible for breaking up the dendritic microstructure. The liquid formation and peritectic temperature were easily determined via the HT-LSCM investigation. Prior to the reverse peritectic reaction, which corresponds to the onset of equilibrium melting, we obtained experimental evidence of the melting of non-equilibrium phases that originated via segregation during solidification. Finally, it was possible to qualitatively study the coarsening mechanisms that occurred in the semi-solid state. Irrespective of the alloy composition (liquid fraction), both Ostwald ripening and coalescence occurred during the isothermal heat treatments. Based on the obtained results, HT-LCSM can be considered a valuable technique for the characterization of thixotropic alloys.

1. Introduction

To date, there have been numerous investigations on the microstructural features of semi-solid alloys. This is because the rheological behavior of the slurry is highly dependent on the morphology of the solid phase [1–3]. If solidification progresses naturally, the nucleated grains develop a dendritic network. Depending on its grain size, this cohesive structure strengthens at only 10 to 20% of solid; consequently, this material will be difficult to deform and prone to cracking [2]. However, when the microstructure is formed by spherical particles immersed into the liquid, the semi-solid material behaves as a thixotropic fluid. In this case, shear causes the viscosity of the material to plummet, thus allowing the manufacture of complex-shaped parts [4,5]. Semi-solid slurries exhibit low viscosity values during processing; however, these values are greater than those of fully liquid metals. This implies that a more controlled flow exists, which consequently lowers the defect levels of the final product [6,7]. Currently, an entire class of processes is based on the thixotropy of semi-solid alloys; these include rheocasting, thixoforming, thixomolding, and many others [8].

Most information on semi-solid microstructures has been obtained using conventional quenching experiments. However, there are several limitations associated with this method when it is used during microstructural examinations. First, the cooling rate must be sufficiently high to prevent or minimize the growth of primary globules; this is a condition that is difficult to attain in large samples. Tzimas and Zavaliangos [9] demonstrated the strong dependence of the measured solid fraction on the distance from the quenched surface. Furthermore, in the case of alloys that solidify in near-single-phase structures with a high solid content, it is usually not possible to identify the former liquid region. Second, coarsening can only be evaluated via statistical techniques. Various samples are typically used to describe the characteristic coarsening behavior; this limits further insight into the coarsening mechanisms.

To overcome these obstacles and obtain detailed descriptions of such semi-solid microstructures, in-situ techniques have been used during real-time observations. More recently, X-ray radiography and tomography have been performed in situ to study the semi-solid coarsening of samples with both dendritic and globular solid phases [10–13]. For instance, Limodin et al. [10] could analyze the local evolution of the microstructure of an Al-Cu alloy treated in the semi-solid state. The authors observed various trends regarding the individual necks between solid particles and found that coarsening

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https://doi.org/10.1016/j.matchar.2018.08.016
Received 22 May 2018; Accepted 9 August 2018
Available online 10 August 2018
1044-5803/

proceeds via a combination of Ostwald ripening and coalescence mechanisms. They also showed that discrepancies exist between the results of in-situ and post-quench experiments, thus highlighting the paramount importance of in-situ studies. Other investigations [14–17] involving X-ray radiography and tomography have also been conducted; in these studies, direct microstructural observations have been used to elucidate the deformation behavior of semi-solid alloys, revealing that they can behave as near-cohesionless granular materials.

High-temperature laser-scanning confocal microscopy (HT-LSCM) is a technique that allows the in-situ and real-time analysis of superficial microstructural features as a function of temperature. Owing to the high-intensity incident light and the presence of the confocal pinhole, which restricts the amount of light that can reach the detector (only light from the focal plane can pass through it), sharp images can be obtained at temperatures as high as 1750 °C [18,19]. HT-LSCM has been successfully used to investigate solidification processes [18,20–22] and solid-to-solid phase transformations [19,23–25]. This technique has been used to elucidate the delta-ferrite-to-austenite phase transformation (peritectic reaction) in steels [26–28]. Based on in-situ observations during peritectic phase transitions, Griesser et al. [29] demonstrated the importance of the presence of a solute diffusion field on the nucleation process that occurs in Fe-C and Fe-Ni alloys. Regarding solid phase transformations, an interesting example can be found in the work of Xu et al. [30]. The thermal cycle that occurs during high-heat-input welding processes was simulated with the aid of HT-LSCM. This facilitated the study of phase transformations. In one study, the effect of the Mg content on the heat-affected zone of steel plates, following welding, was evaluated. Some examples have been given here to show the versatility of this technique and its effectiveness regarding the study of high-temperature events. However, only a few studies have used HT-LSCM to investigate alloys for semi-solid processing [31–34]. Recently, Benati et al. [34] characterized the phase transformations that occur during the reheating to the semi-solid state of a gray-cast iron. Although the authors could not measure the liquid fraction as a function of temperature, they succeeded in observing the onset of melting and the detachment of solid grains by the liquid phase.

Since a study by Zhao et al. [35] was published, Ti-Cu alloys have been increasingly considered for thixoforming [36–40]. The term thixoforming can be used to describe a general semi-solid processing route in which the initial raw material is solid. Moreover, it also refers to a specific semi-solid processing technique in which a solid material is vertically heated until it consists of 30–50% liquid, and is subsequently inserted into a die [8,41]. Considering the latter meaning, in previous investigations [39,40], Ti-Cu binary alloys were selected to achieve liquid fractions in the aforementioned range at relatively low temperatures. These alloys were reheated and treated in the semi-solid state for up to 10 min. The results showed that globular microstructures and low coarsening rates could be achieved; these are both desirable characteristics regarding this process. These microstructural studies were entirely based on rapid-quenching experiments and hence, in-situ observations in real time and at temperature provides new opportunities to study melting, globular microstructure formation, and coarsening mechanisms in these alloys. In response, we have used high-temperature laser-scanning confocal microscopy (HT-LSCM) to conduct an in-depth investigation on Ti-Cu alloys during reheating, partial melting, and isothermal heat treatments in the semi-solid state. Moreover, we will discuss the advantages and limitations associated with employing this technique for the characterization of alloys intended for thixoforming.

2. Experimental Procedure

High-purity elements were arc-melted, under an argon atmosphere, to form ingots with compositions of Ti-(25,27,29)Cu (wt%). These ingots were then homogenized at 950 °C for 24 h, furnace cooled, and hot swaged at 900 °C followed by air-cooling until cylindrical bars with a diameter of 11.5 mm were obtained [39,40]. A part of each cylindrical bar was machined to a final diameter of 9.8 mm, whereas another part was machined to a final diameter of 3.6 mm.

In-situ observations were performed using a high-temperature laser-scanning confocal microscope (Lasertec Corp.) equipped with an infrared furnace employing a 1.5-kW halogen lamp, which was used to heat the samples. More detailed descriptions of the construction and operation of this microscope are well-documented and need not be repeated [18,19]. All the experiments were conducted under a high-integrity argon atmosphere. The oxygen level within the furnace was measured by using a RAPIDOX 2100 sensor, and maintained below 10^{-13} ppm in order to avoid oxidation of the specimen surface. The samples were heated within alumina crucibles with diameters of 4.3 or 10 mm, respectively. A thin layer of yttrium oxide was used to prevent reactions between the sample and the crucible.

Samples with a diameter and thicknesses of 3.6 mm and 1 mm, respectively, were polished on one side, placed into the crucible and reheated to the semi-solid state. A heating rate of 100 °C/min was applied from room temperature to 900 °C. Between 900 °C and 950 °C, the heating rate was reduced to 20 °C/min. Subsequently, from 950 °C to the onset of melting, an even slower heating rate of 2 °C/min was used. It is noteworthy that a hold of 1 min was performed at 200 °C to eliminate some moisture from within the furnace. This hold is a standard procedure and it was performed during all the experiments; however, it did not affect any of the results described here. The lower heating rate prior to melting was intended to allow observation of the transformations and to decrease the temperature difference between the specimen surface and the measuring point (a B-type thermocouple was welded onto the platinum crucible holder). A variety of thermal cycles were also applied, which will be outlined below.

When investigating molten alloys, there is an intrinsic problem associated with the use of HT-LSCM techniques that employ conventional small samples (diameter and thickness of around 4 mm and 1–2 mm, respectively). Owing to the shallow depth of focus in confocal microscopy, high quality images are difficult to be obtained because of the formation of a liquid meniscus. To overcome this impediment, the so-called ‘concentric solidification technique’ was developed by Reid, Phelan, and Dippenaar [18] at the University of Wollongong. By this technique, cylindrical samples of 9.8 mm diameter and about 0.25 mm thick are used and because the thermal profile within the furnace has an inverted ‘V' shape, a radial thermal gradient is established within the sample, which allows a liquid pool to be formed in the center, supported by an outer solid rim. The meniscus problem is overcome, a much larger area is kept in focus and the image quality is significantly improved. The concentric solidification technique coupled with HT-LSCM allows for the formation of a relatively controlled liquid pool within the sample. Thus, this approach was used to determine the semi-solid coarsening behavior of the Ti alloys employed in this study. However, during these experiments, the center of the sample is not fully liquid, but is maintained in the semi-solid state. A heating rate of 100 °C was employed from room temperature to 850 °C. The heating rate was then decreased to 20 °C/min between 850 °C and the onset of melting. Under equilibrium conditions, the melting of these Ti-Cu alloys commences with the reverse peritectic reaction [42,43]. Accordingly, an isothermal heat treatment at a temperature close to the peritectic temperature was performed for up to 1 h. There are two main reasons for treating the samples close to the peritectic temperature. Firstly, these alloys present a wide solidification interval, i.e., if the temperature is much higher than the peritectic temperature, the outer solid rim starts to melt. Secondly, in larger samples, the temperature difference between the observation field and the temperature measuring point is greater and hence, the temperature of the reverse peritectic transformation was taken as a reference temperature to calibrate the experimentally measured temperatures.

Images were captured from the videos that were recorded throughout the experiments. Where applicable, image analysis was