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Microstructural control of metastable phase in rapidly solidified Ti-45Al-2Cr-2Nb alloy

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Rapid solidification processing (RSP) allows an alloy melt to be undercooled below the equilibrium melting point. It is possible for a primary metastable phase to solidify from a melt undercooled below the melting point of the equilibrium phase as the large undercooling generated in the melt favors nucleation of a metastable phase preferentially over stable phase [1, 2]. The major emphasis of RSP so far has been on improving the rate of heat removal from the melt or the solidification material, which makes it difficult to clarify the phase separation mechanism in the rapidly solidified materials. Thus, various, even contrary, results on the phase evolution behavior in rapidly solidified alloys have been reported (see e.g., [3–6]).

Microstructural evolution in a non-equilibrium solidification process depends critically on the melt undercooling of competing phases, and the role of undercooling on formation of metastable phases can be investigated by high undercooling techniques [7–9]. In this paper the phase evolution relationship during RSP will be explored systematically and efforts will be made to explore the possible design of phase composition in rapidly solidified alloys.

A Ti-45Al-2Cr-2Nb alloy was used for this investigation as it is the most promising representative of TiAl-based alloys for a light, strong and chemically stable materials for structural application at high temperature [10, 11]. High-purity aluminum, titanium, chromium and niobium (>99.9%) were taken in the required proportions to form an alloy with a stoichiometry of Ti-45Al-2Cr-2Nb. The process of melting was carried out in a vacuum arc furnace under a flowing high pure argon atmosphere, to produce a button of approximately 4 cm in diameter that was remelted four times. The arc-melted buttons were homogenized in a vacuum heat treatment furnace at 1273 K for 80 h in order to achieve complete composition homogenization.

A typical transmission electron microscopy of the homogenized Ti-45Al-2Cr-2Nb alloy is illustrated in Fig. 1. It was composed of the lamellar structure ($\alpha_2 + \gamma$), with the black α_2 phase interspersed between many white γ plates. The lamellar structure was transformed from primary phase α due to the solid-transformation in the subsequent cooling process. This observation of α as primary phase agrees well with the recently revised phase diagram of Ti-45Al-2Cr-2Nb [12].

Figure 1 Typical microstructure of Ti-45Al-2Cr-2Nb homogenized at 1273 K for 80 h.

The undercooling experiments were carried in an electromagnetic levitation melting apparatus manufactured by Edmuld Buhler Co. The working chamber was initially evacuated to about 10^{-9} mbar, then back-filled with high purity argon gas (>99.999%). For the purpose of deactivating heterogeneous nucleation sites, each sample was cyclically superheated by 300 K for five minutes. An infrared pyrometer recorded the thermal behavior of the sample [13]. The melting temperature and undercooling on cooling could be obtained from the comparison with the absolute temperature recorded by the standard thermocouple.

The maximum achieved undercooling of Ti-45Al-2Cr-2Nb was up to 252 K and the phase α was detected as the primary phase over the entire undercooling range. Attempts to obtain bulky metastable γ phase from the undercooled Ti-45Al-2Cr-2Nb were made by manually changing the nucleation competition among the stable and metastable phases. In this process a triggering needle of metastable phase γ was employed

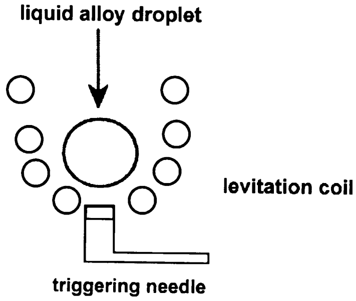

Figure 2 Schematic diagram of metastable materials preparation from the undercooled melts.

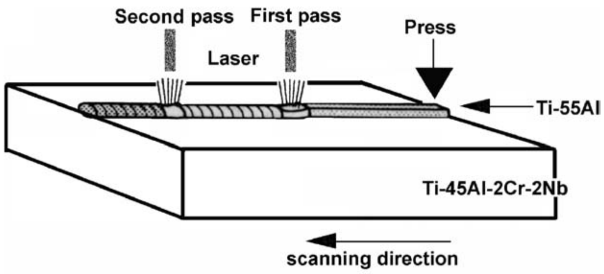

Figure 3 Schematical diagram of achieving the surface with metastable γ phase.

to get metastable structure from the undercooled alloy melt. The schematic diagram of preparation of bulky metastable materials from the undercooled melt is shown in Fig. 2. When the melt of Ti-45Al-2Cr-2Nb alloy was effectively undercooled to a large degree by several heating and cooling cycles, a triggering needle of $Ti_{44}Al_{56}$ alloy with single-phase γ structure was put right into the levitation coil. After another heating process, the power of the high frequency generator was turned down slowly. When it cooled and arrived at a desired undercooling value, the power was immediately shut off and the levitated sample dropped on the needle and started to solidify. By this method the competitive nucleation of stable (α) and metastable phases (γ) in the undercooled Ti-45Al-2Cr-2Nb alloy melt was overcome when the undercooling of the melt was larger than a critical value, 54 K, and the bulky sample with metastable γ phase was successfully prepared [14].

In a rapid solidification process, phase selection from highly undercooled melts is determined by the kinetics of nucleation and growth for either the equilibrium solid or one of several possible metastable phases. Thus, if the nucleation process can be controlled in some way, it might be possible to form a metastable phase [15].

The application of the undercooling technique was mainly obstructed by the poor availability of high undercooling. However, laser resolidification process tends to be much more convenient to explore the influence of nucleation on achieving a metastable surface because of the intimate contact of the surface layer with the substrate. Here the laser resolidification experiment was carried in a Rofin-Sinar 850 continuous wave CO_2 laser. The specimen was polished down to 1000 grade SiC emery paper and thoroughly cleaned in methanol prior to the laser treatment. The melted pool was shielded to prevent oxidation by blowing high-pressure helium over the surface during the laser treatment.

Following the above experimental observation in undercooling experiments, the selection of stable (α) and metastable (γ) phases was attempted in Ti-45Al-2Cr-2Nb alloy melt by changing the outer nucleation substrate. Fig. 3 shows systematically the system to produce a metastable γ surface in Ti-45Al-2Cr-2Nb alloy. A rod-like sample of $Ti_{45}Al_{55}$, of phase γ, was placed on a substrate of Ti-45Al-2Cr-2Nb alloy. Laser resolidification was carried out to embed the melt on the substrate. With the first laser path, however, the thermal contact with the substrate was not always effective. As a reverse process, the metastable phase retained at room temperature can be heated to its melting point and melted, by-passing any solid-state phase transformation or decomposition due to fast heating rate. The second laser melting of the first track allowed the growth of metastable primary phase from the bottom of the sample, that is, metastable dendrites, of primary phase γ, formed in the surface of Ti-45Al-2Cr-2Nb alloy.

The microstructure thus obtained is presented in Fig. 4. The resolidified layer is composed of white dendrites of phase γ (Fig. 4a), and a metastable layer of γ phase formed on the surface of fully lamellar Ti-45Al-2Cr-2Nb alloy (Fig. 4b). This kind of microstructure design of metastable phase γ would greatly improve the service performance of fully lamellar TiAl-based alloys. Fig. 5 illustrates the TEM analysis of the modified surface in the laser resolidified Ti-45Al-2Cr-2Nb alloy. As seen from the bright field image (Fig. 5a), there are some dislocations distributed in the white matrix,

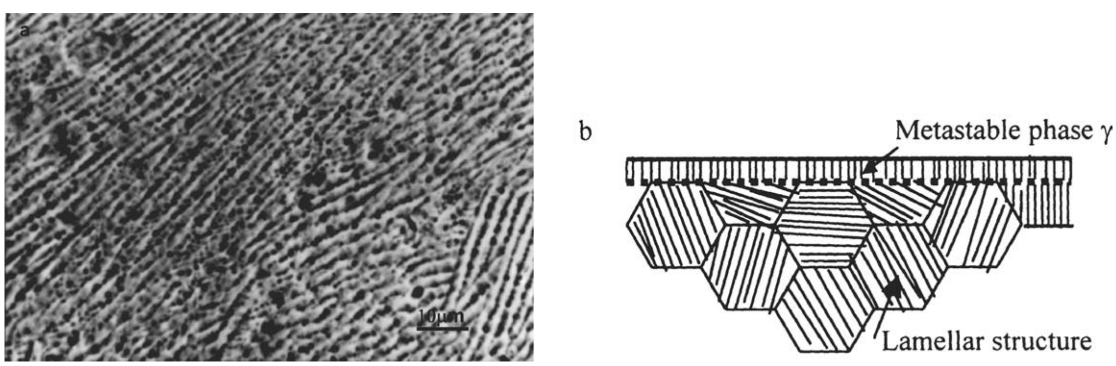

Figure 4 Microstructural morphologies of the modified surface in the laser resolidified Ti-45Al-2Cr-2Nb alloy: (a) local SEM image and (b) schematic interpretation of the transverse section.

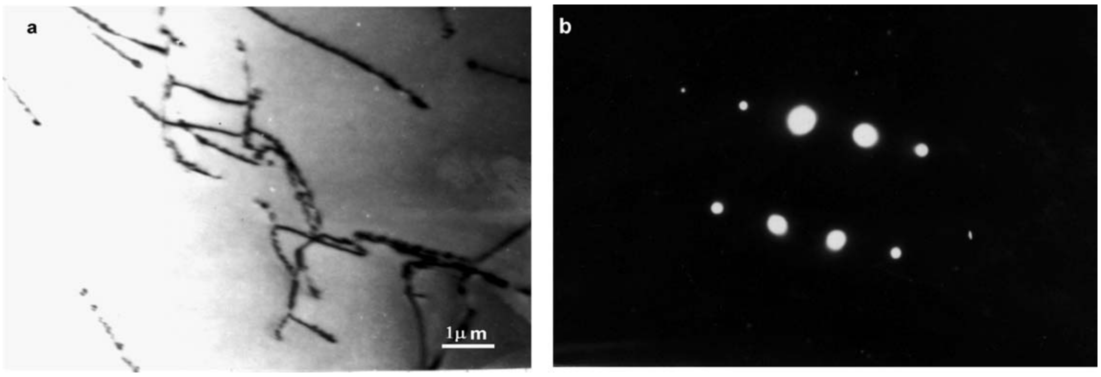

Figure 5 TEM analysis of the modified surface in the laser resolidified Ti-45Al-2Cr-2Nb alloy: (a) bright field image and (b) selected area diffraction patterns of gamma phase.

which was proven to be metastable γ phase according to the selected area diffraction patterns (Fig. 5b).

In summary, a surface treatment method was developed to control the phase composition and/or crystalline structures. A metastable structure (γ phase) was successfully introduced on the surface of a fully lamellar Ti-45Al-2Cr-2Nb alloy.

Acknowledgments

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