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To cite this article: O Shuleshova *et al* 2009 *J. Phys.: Conf. Ser.* **144** 012118

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Phase selection in undercooled Ti-Al-Nb melts

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Abstract. Non-equilibrium solidification of undercooled Ti-Al-Nb melts with compositions around the border of the α -Ti/ β -Ti phase domains are investigated using the electromagnetic levitation technique. Detailed thermal histories of rapid recalescence processes acquired with a high time-resolution by a fast responding photo-diode are compared to *in situ* analyses of the solidified structures by using energy dispersive x-ray diffraction. The primary formation of the β -Ti phase is observed independent on the undercooling level up to a maximum undercooling of 300 K at compositions near the border of the α -Ti regime. On cooling the primary phase completely transforms into the α -Ti phase from which γ -TiAl precipitates at considerably lower temperatures.

1. Introduction

Containerless processing methods, such as electromagnetic levitation (EML), are powerful techniques to study the influence of melt undercooling on phase selection during solidification of metallic melts. With increasing undercooling level below the metastable liquidus temperature the nucleation and growth of metastable phases could be preferred by thermodynamics and/or kinetics instead of stable solids. Such metastable phases are often subjected to a fast transformation into the stable counterpart. Commonly an *in situ* thermal analysis is used to get the information on structural transformations via a thermal response to the heat released or absorbed during the phase transition. One technique to resolve different phase transformations occurring within short times during the rapid solidification is thermal analysis by observation of the sample surface using a fast responding photo-diode. This approach has been applied to study the metastable phase formation in near-equiatomic alloys of the Ti-Al binary system [1], in particular for alloys solidifying within the stable β -Ti and α -Ti regime.

In present work the effect of undercooling on phase selection is studied for the Ti-Al-Nb ternary system [2]. Alloys of β solidification type in the vicinity of the α primary domain are considered. The EML technique is combined with the photo-diode method and *in situ* diffraction of synchrotron radiation.

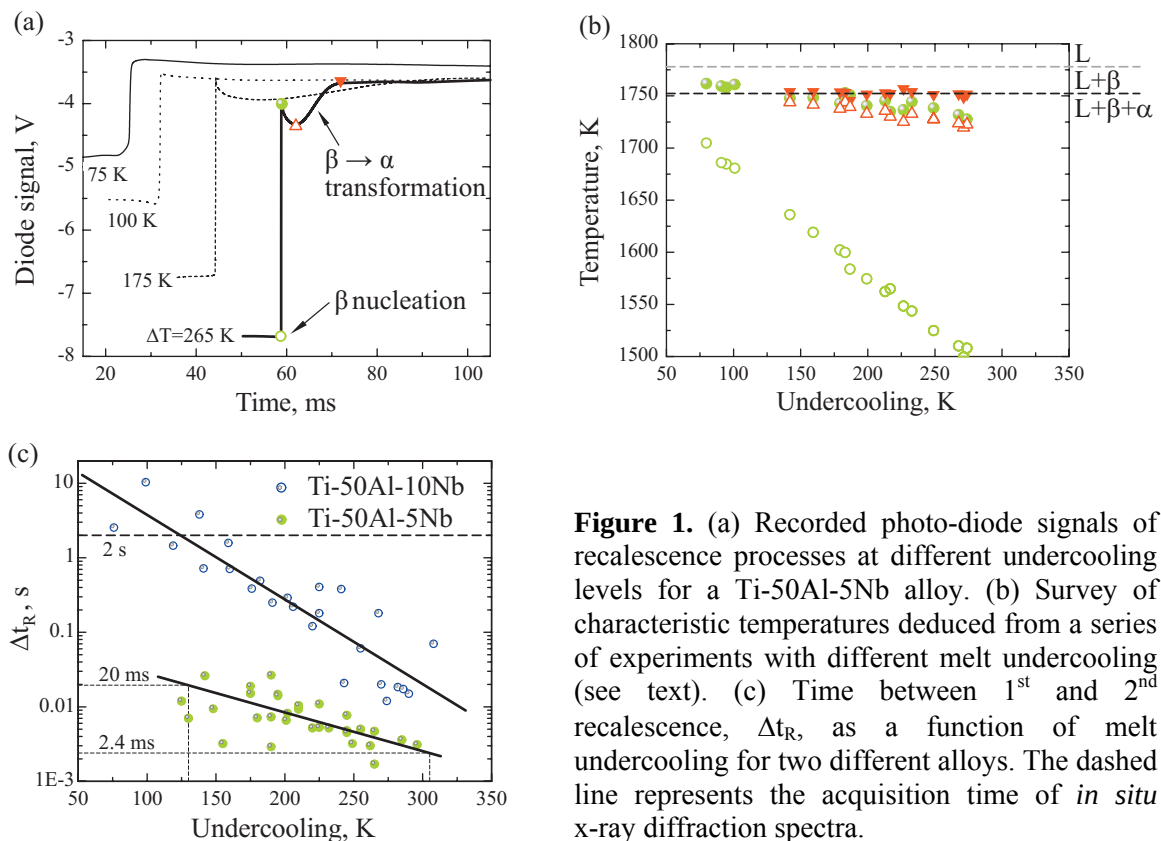
2. Experimental methods

Samples were prepared from the elements Ti, Al and Nb with 99.99 % purity by induction melting. For deep melt undercooling the EML technique was utilized, which allows containerless processing of the melts under high-purity conditions. The EML apparatus detailed by Löser et al. [3] was used. The levitated samples were repeatedly melted, heated to temperatures above the liquidus temperature, kept

at high temperature for purification and then cooled by a gas stream below liquidus temperature. Solidification was stimulated at specific undercoolings by touching the liquid droplet with a W needle. Detailed thermal histories of rapid recalescence processes were acquired during experiments by a fast responding Si photodiode with sampling rates of 2 MHz along with a conventional two-colour infrared pyrometer (time resolution 10 Hz). The *in situ* determination of the structure of solidified phases was accomplished by combination of a specially designed electromagnetic levitator with the high-energy white beam diffraction setup available at the beamline ID15A of the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. An experimental setup analogous to that described in reference [4] was used. The application of high-energy radiation allows for diffraction studies in transmission of bulk samples approx. 1.6 g in mass. The diffracted intensity is measured with an energy-dispersive Ge detector under a constant scattering angle of $2\theta = 2.5^\circ$. The minimum acquisition time for recording spectra of reasonable quality was 2 s. During this series of the levitation experiments the temperature of the sample was measured using a one-colour pyrometer at a sampling rate of 20 Hz.

3. Results and discussion

Two alloys, Ti-50Al-5Nb and Ti-50Al-10Nb, of β -Ti primary solidification type situated near the border to the α -Ti primary phase domain [2] were investigated. A detailed analysis of typical thermal histories obtained during undercooling experiments has been reported previously [5]. In the temperature-time profiles measured with a pyrometer at 0.1 s time resolution several thermal events could be distinguished. In general, as the sample reaches a certain temperature T_N below liquidus temperature T_L the nucleation (spontaneous or induced) triggers a rapid non-equilibrium solidification process. The fast quasi-adiabatic release of latent heat of fusion causes a sudden rise of the sample temperature, called recalescence. For both alloys the first recalescence event was attributed to the primary solidification of β -Ti phase, during which the temperature increases up to a maximum temperature between the liquidus and the solidus temperature. The subsequent $\beta \rightarrow \alpha$ transformation, expected from the phase diagram [2] was judged to release not enough heat to be detected as a well



defined thermal event on the pyrometer curve. The second thermal response observed at lower temperatures was attributed to the $\alpha \rightarrow \gamma$ solid state phase transformation.

The analysis of recalescence events at different undercooling levels for Ti-50Al-5Nb alloy presented in figure 1a revealed the more complex solidification behaviour. As the undercooling increases a smooth transition from one-step to two-step recalescence occurs. A double recalescence indicates that the phase formed during the 1st event undergoes a phase transformation causing a second temperature rise. An overall analysis shows that the maximum temperature reached at 1st recalescence, T_{R1} , monotonically decreases with rising undercooling, while the temperature, T_{R2} , reached after the 2nd recalescence is constant and well below the liquidus temperature. Moreover, the continuity observed for T_{R1} implies that the primary phase remains the same (β -Ti) for all undercooling levels. Thus, the double recalescence event observed does not indicate a metastable phase formation but results from a fast transformation of primarily formed β phase into α if the sample approaches the L+ β + α ternary phase field, where the peritectic reaction β +L $\leftrightarrow$ α sets in [2]. The time between 1st and 2nd recalescence, Δt_R , strongly depends on the melt undercooling and alloy composition (figure 1c) [5].

To confirm this conclusion, *in situ* x-ray diffraction studies on the phase selection as a function of the melt undercooling were accomplished. In figure 2 results of such experiments are shown for the Ti-49.5Al-4.8Nb alloy. Prior the recalescence event the sample is in a molten state represented by diffuse intensity maxima of the EDXD pattern. As the recalescence sets in distinct Bragg reflections emerge from the diffuse intensity, indicating the nucleation and growth of a solid phase. At moderate melt undercooling $\Delta T = 130$ K they are identified as (100) (not visible on this scale) and (110) reflections of the β -Ti phase (figure 2a). The subsequent α -Ti phase reflections indicate the onset of $\beta \rightarrow \alpha$ transformation, which corresponds to the 2nd recalescence event in the thermal data with high timely resolution. The apparent time, ~ 0.5 s, during which solely the β phase exists is quite high compared to that expected (~ 20 ms from figure 1c). This might result from a small volume fraction of

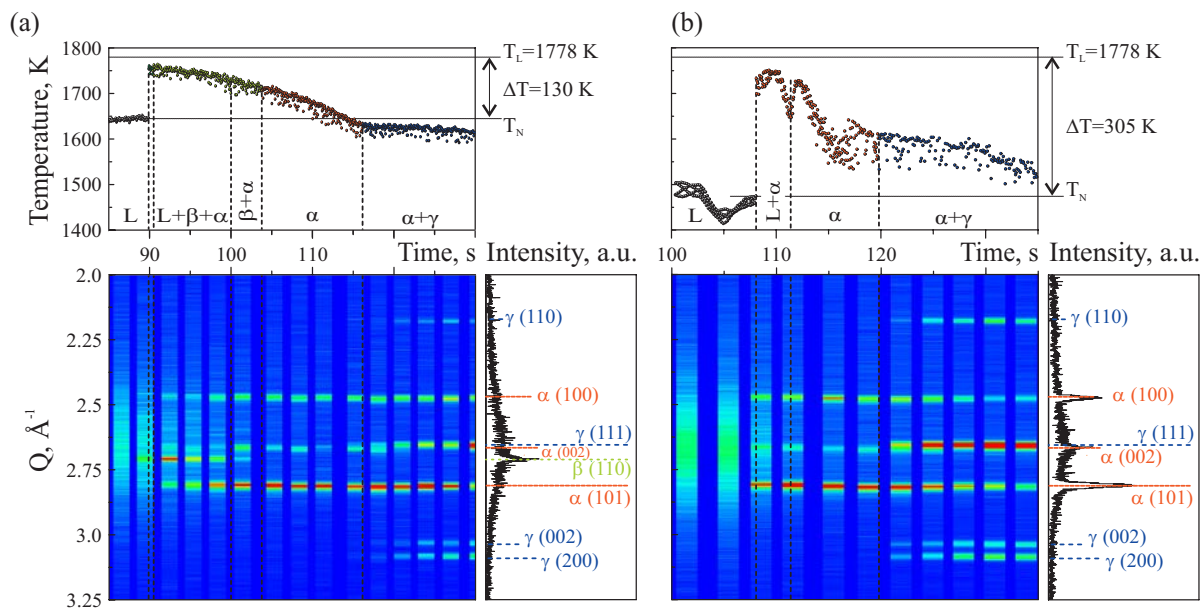


Figure 2. Time evolution of the temperature and crystal structure for undercooling cycles on a Ti-49.5Al-4.8Nb alloy. The temperature-time profile (top) is synchronized in time with the EDXD spectra (bottom). On the intensity map spectra appear as narrow stripes with lengths reflecting the 2 s acquisition time separated by the time required for data transfer. The darkness of the stripes in the intensity map indicates the intensity of the Bragg reflections. The EDXD spectrum where the first reflections of crystalline phases appeared is plotted together with the positions of the Bragg peaks of all relevant phases (bottom right). (a) β -Ti primary phase at moderate undercooling $\Delta T = 130$ K. (b) The α -Ti phase is the first one observed during solidification at deep undercooling $\Delta T = 305$ K.

the α phase formed at the beginning of the transformation that is below the detection limit. On further cooling a three phase field ($L+\beta+\alpha$) indicates the peritectic reaction range. In the experiment cycle shown the peritectic reaction is incomplete and the rest of the β -Ti phase undergoes a solid state transformation. Then the sample passes through the α single phase field until γ -TiAl phase precipitates at temperatures around $T = 1625$ K.

In contrast, at high melt undercooling $\Delta T = 305$ K, the (100), (002) and (101) reflections of α -Ti are observed immediately in the first diffraction spectrum (figure 2b). At the same time double recalescence unambiguously indicates a transformation event, proving that the α phase is not the primary phase but a transformation product. The absence of the primary β phase reflections is expected, because for the given undercooling the $\beta \rightarrow \alpha$ transformation sets in after ~ 2.4 ms (figure 1c), which is one order of magnitude shorter than at $\Delta T = 130$ K. After recalescence the sample passes through the $L+\alpha$ two-phase field until complete solidification. Subsequently the γ phase is formed.

The time between first and second recalescence in the order of ms observed for deeply undercooled Ti-Al-Nb melts is two orders of magnitude higher than that observed in binary Ti-Al melts [1]. The overall recalescence characteristics resemble that of the metastable β phase within stable α regime reported for the binary system. Such speeding-up of the $\beta \rightarrow \alpha$ transformation is explained by the opening of the ternary $L+\beta+\alpha$ phase field with addition of Nb. Since the maximum temperature reached during primary recalescence decreases with increasing undercooling it approaches the $L+\beta+\alpha$ range where small undercooling is sufficient to drive the transformation resulting in the fast double recalescence event.

4. Conclusions

Combination of *in situ* x-ray diffraction experiments on levitated samples with *in situ* studies using a fast photo-diode based thermal analysis allowed us to deduce unambiguously the phase selection during the non-equilibrium solidification from undercooled Ti-Al-Nb melts. No change in primary phase selection of melts undercooled up to 300 K is observed within the equilibrium β -Ti phase domain. On cooling the primarily solidified β -Ti completely transforms into the α -Ti phase from which γ -TiAl precipitates at considerably lower temperatures. However, at compositions near the border of the α -Ti regime the subsequent $\beta \rightarrow \alpha$ transformation occurs within 10^{-3} s, while the sample is still in the semi-solid state. The transformation time of the equilibrium phase becomes comparable with the typical timescale of life times observed for metastable phases.

5. Acknowledgements

The authors thank T. Buslaps, H. Hartmann, T. Volkmann, and N. Mattern for support at the beamline and D.M. Herlach for fruitful discussions. This work was funded by the European Commission within the IMPRESS Integrated Project (FP6-500635-2) and by ESRF Experiment-No. MA-179.

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