Effect of convection on the dendrite growth kinetics in undercooled melts of D2 tool steels

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Abstract Rapid solidification of D2 tool steel is investigated experimentally using the electromagnetic levitation technique under terrestrial and reduced gravity conditions. The microstructures of samples covering a broad range of undercoolings (40 K ≤ ΔT ≤ 280 K) are analysed. At low undercooling coarse grained dendritic microstructure is observed, while at higher undercoolings this dendritic feature disappears in favour of a grain refined equiaxed structure. In the latter case, the eutectic carbides are more evenly dispersed throughout the microstructure. The sample solidified in microgravity during parabolic flight experiment exhibits only a few very large grains with twinning relationship. This highlights the effect of convection on grain refinement in this system.

1. Introduction

D2 tool steels are widely used in industry because of their good wear and abrasion properties [1]. This is due to the high volume fraction of carbides that precipitate during the eutectic reaction. The wear resistance can be further improved by reducing the size and evenly distributing carbides [4]. Therefore, a rapid solidification technique is used on D2 tool steel to refine the microstructure. In addition to the cooling rate, the undercooling ΔT prior to solidification has a significant effect on the microstructure during solidification of metals. However, systematic experimental work on the rapid solidification of D2 tool steels is still lacking.

In this study, electromagnetic levitation (EML) is applied to droplets of D2 tool steel. This containerless technique enables direct measurement of the undercooling prior to solidification and of the velocity of the growth front. Comparison between results obtained under terrestrial and reduced gravity conditions allows the investigation of convection effect in dendrite growth dynamics and in the evolution of the microstructure.

2. Experimental

The D2 tool steel is a high carbon, high chromium ferrous alloy with Fe-1.55C-11.8Cr-0.40Mn-0.80Mo-0.80V-0.30Si (all in wt.%). The corresponding pseudo-binary phase diagram is shown in Figure 1 (calculated using ThermoCalc software and the TCFE6 data bank), with the dotted line highlighting the carbon content of D2 steel. Upon cooling, solidification starts with the crystallization of austenite followed by the eutectic decomposition of the interdendritic liquid into austenite and carbide \((L\rightarrow \gamma + (\text{Cr,Fe})_7\text{C}_3)\). At room temperature, the microstructure consists of ferrite and \((\text{Cr,Fe})_7\text{C}_3\). However, during rapid solidification of tool steels metastable supersaturated austenite can be retained in the microstructure [3].
For in-situ studies of deeply undercooled D2 tool steel melts the electromagnetic levitation technique (EML) is used as schematically shown in Figure 2. This containerless technique provides a large range of undercoolings prior to solidification and allows direct observation of the solidification process. The release of latent heat during rapid solidification leads to a visible contrast between the solid and the undercooled liquid (the former appearing brighter than the latter). This enables the recording of the solidification front by a high-speed video camera. The temperature is measured contactless by an infrared-pyrometer. High purity 6N He is used to cool down the sample. Detailed information on the EML technique can be found in [4].

3. Results and discussion

Figure 3 shows high-speed video snapshots of electromagnetic levitated samples solidified at different undercoolings $\Delta T$. At low $\Delta T$ a single coarse equiaxed dendrite is growing through the undercooled sample (a). At higher $\Delta T$ several fine dendrites originating from a single nucleation point can be seen (b). At very high $\Delta T$ the front becomes spherical with no observable dendritic features (c). As expected, the growth velocity of the front increases with the undercooling.

Figure 3. High-speed video images of electromagnetic levitated samples solidified at different undercoolings $\Delta T$. The dark grey area is the undercooled liquid. The light grey region corresponds to the growing solid, which appears brighter due to the release of latent heat during recalescence. a. $\Delta T = 45$ K, $v = 0.03$ m/s. b. $\Delta T = 179$ K, $v = 1.3$ m/s. c. $\Delta T = 272$ K, $v = 8.4$ m/s. A transition from coarse to fine dendrites to spherical front is observed.

Micrographs of the microstructures of these three samples are presented in Figure 4. In all cases, primary austenite and interdendritic austenite + carbide eutectic can be identified. Rapid solidification
of D2 in EML completely suppresses the formation of ferrite and martensite. At \( \Delta T = 45 \) K a classic coarse grained dendritic microstructure is observed, while at higher undercoolings this dendritic feature disappears in favour of a grain refined equiaxed structure. It is assumed that grain refined microstructures were developed by the faster growth dendritic velocity at higher undercoolings and possibly by dendrite fragmentation after recalescence [5]. High undercoolings give rise to much finer microstructures, with the cell spacing decreasing from about 10-30 µm at \( \Delta T = 179 \) K to 5-10 µm at \( \Delta T = 272 \) K. This results in a better dispersion of the \((\text{Cr,Fe})_7\text{C}_3\) carbides throughout the microstructure.

Comparing the micrographs of samples b and c with the corresponding high speed video recordings of solidification, there appears to be no notable change in microstructure with regards to the solidification front morphology apart from the cell size. This suggests that the spherical front depicted in Figure 3c is composed of very fine dendrites whose features are too small to resolve on the recording.

![Figure 4. SEM micrographs of cross sections of D2 samples solidified at \( \Delta T = 45 \) K, 179 K, and 272 K. Dendrites or cells of austenite can be observed, surrounded by the eutectic.](image)

Figure 4 shows electron back-scattered diffraction maps (EBSD) and the corresponding pole figures for samples b and c. Both samples exhibit an equiaxed polycrystalline microstructure. At \( \Delta T = 179 \) K the grain size ranges from 100 to 500 µm while at \( \Delta T = 272 \) K the range is 10-50 µm, i.e. the highly undercooled sample shows a stronger grain refinement. As the measured growth velocity is high, the dendrites are expected to be very slim. Thus, fragmentation of the primary trunks or secondary arms would be easier to achieve.

![Figure 5. EBSD maps and corresponding pole figures of cross sections of D2 samples solidified at \( \Delta T = 179 \) K and 272 K respectively.](image)
In order to observe the possible influence of convection on the solidification of levitated D2 tool steel, parabolic flight experiments were performed under reduced gravity conditions using the TEMPUS facility [6]. In µg-EML the fluid flow is about 0.05 m/s, compared with 0.3 m/s during ground experiments [7]. Figure 6 shows the micrograph of a cross section of a sample solidified in such conditions at an undercooling of $\Delta T = 164$ K. At first glance the microstructure appears similar to that of the sample solidified under 1g conditions at $\Delta T = 179$ K. Both the primary austenite and the austenite + (Cr,Fe)$_7$C$_3$ carbide eutectic are observed. Furthermore, the cell size of the primary phase ranges also from 10 to 30 µm. However, diffraction results are strikingly different. The EBSD map in Figure 7 shows only two different grains, one large grain covering the majority of the cross section (about 1 mm in size) and one small grain in the lower left part. Interestingly, these two grains have a twin relationship with respect to each other. The common $\langle 111 \rangle$ direction and the trace of the $\{111\}$ twin plane containing the three common $\langle 110 \rangle$ directions are highlighted on the pole figures. It is not known if solidification started with one single grain with subsequent stacking faults or if twinning occurred at nucleation. EBSD analysis performed on a cross section deeper into the sample (not shown here) revealed three grains with twin relationships, two of them being the same grains as in Figure 7. The size of the largest grain is about 2.3 mm but is probably larger as the EBSD map only covered a portion of the cross section. Such large grains have never been observed in D2 samples solidified in 1g conditions at undercoolings of the same magnitude. This clearly indicates that convection in the undercooled liquid during EML solidification plays a major role in grain refinement in this system. Whether fluid flow promotes fragmentation of the dendrites via mechanical shear or local remelting due to solute redistribution remains however unclear.

![Figure 6. SEM micrograph of a cross section of a D2 sample solidified at $\Delta T = 164$ K in µg.](image)

![Figure 7. EBSD map and pole figures of a cross section of a D2 sample solidified at $\Delta T = 164$ K in µg. The common $\langle 111 \rangle$ direction and the trace of the $\{111\}$ twin plane are highlighted.](image)

4. Conclusion

In this study, droplets of industrial D2 tool steel were solidified in EML over a wide range of undercoolings. Samples solidified at high undercooling exhibit a fine microstructure with strong grain refinement. This results in a better dispersion of the eutectic carbides as at lower undercoolings. Rapid solidification should thus prove to be a good way to improve the mechanical properties of this alloy.

EBSD analysis showed large differences in the microstructure between droplets solidified on the ground and in microgravity conditions. While the former ones are polycrystalline with many different grains, the sample solidified in µg shows very few much larger grains with twinning relationships. This indicates that fluid flow has a strong influence on grain refinement in this system.

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