Comparison of microstructures developed during solidification of undercooled tool steel in levitation and on a substrate

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Abstract. In this paper, the microstructures developed in highly undercooled samples from Cr-Mo-V tool steel of ledeburite type during non-equilibrium solidification in levitation and on a copper substrate are compared. The microstructures in spherical samples solidified during levitation are dependent on the level of undercooling reached prior to spontaneous nucleation. In the whole volume of a single rapidly solidified sample, microstructures are quasi-homogeneous. Rapidly solidified splat samples were prepared using copper substrate solidification from the melt with various initial undercooling. As an example, the microstructure in the sample solidified from the initial undercooling of 100 K with morphologically different microstructural regions is presented. Carbides of solidification origin with different size, morphology and location were found in samples solidified in levitation and on a substrate as well.

1. Introduction

High chromium alloyed tool steels (usually with 12 wt. % Cr, other carbide forming elements and high carbon content) are known as ledeburite steels. Hard carbides of eutectic origin are typically present in these steels [1, 2]. Size, morphology and distribution of carbides of solidification origin have a crucial influence on their technological and utility properties. Continual or quasi-continual intergranular carbide phases and eutectic colonies represent the main cause of brittleness of high alloyed tool steels of ledeburite type. Solidification of Cr-Mo-V ledeburite steel in quasi-equilibrium conditions starts by austenite formation and it is finished by two eutectic reactions resulting in the development of morphologically different MC and M7C3 carbides [3, 4]. In the RS powder of Cr-Mo-V hypoeutectic tool steel of ledeburite type variable solidification microstructures were observed [4, 5]. Several different types of microstructures were identified also in quasi-spherical samples from this type of steel prepared by solidification in the conditions of electromagnetic levitation [6, 7]. In this paper, microstructures developed in undercooled samples from Cr-Mo-V hypoeutectic tool steel during rapid solidification using two types of levitation experiments were investigated. The method of rapid solidification after spontaneous nucleation of undercooled samples in levitation was focused on the determination of the maximum achievable level of undercooling prior to solidification of this type of steel and on the examination of the influence of initial melt undercooling on the main microstructural features. Microstructures obtained by rapid solidification of samples in levitation after spontaneous nucleation were compared with microstructures developed in samples dropped down and solidify on a copper substrate with defined melt undercooling.
2. Experimental
The tool steel of ledeburite type with the nominal chemical composition of 2.3% C, 12.5% Cr, 1.1% Mo, 4.0% V and balance Fe [wt. %] was produced in industrial conditions. Rapidly solidified samples from this steel were prepared using electromagnetic levitation. A comprehensive description of the levitation facility is given elsewhere [7]. Samples were several times heated and melted in the vacuum chamber and then cooled by inert gas in levitation or splat quenched on a copper substrate. The weight of levitated samples was about 0.8 g corresponding to quasi-spheres of 5.6 – 6.4 mm in diameter after solidification in levitation or splats with diameter approximately 10 mm after substrate solidification. In order to characterize the microstructure features and to identify the phases presented in the samples, light microscopy, scanning electron microscopy and X-ray diffraction analysis were used.

3. Results and discussion

3.1. Microstructures in spontaneously solidified samples in levitation
During levitation experiments, the melt undercoolings from 50 K to 290 K below the equilibrium liquidus temperature were achieved. The spontaneous solidification of samples started the most frequently at the undercooling between 200 K and 250 K. The microstructures of quasi-spherical samples rapidly solidified in levitation were uniform in the whole volume. This can be documented by the cross-section of the spherical sample undercooled during levitation experiment by $\Delta T = 135$ K (figure 1a). The microstructure of this sample consists of metastable austenitic dendrites and two types of eutectics located in interdendritic regions (figure 1b). Details in figures 2a, b illustrate the size, morphology and distribution of $\text{M}_7\text{C}_3$ and $\text{M}_4\text{C}_3$ based eutectics.

![Figure 1](image1.png)

**Figure 1.** Microstructure of the spherical sample spontaneously solidified in levitation with the initial undercooling of $135$ K, cross-section of the sample (a) and the detail of typical microstructure (b).

![Figure 2](image2.png)

**Figure 2.** Size, morphology and distribution of carbides in the samples spontaneously solidified with the initial undercooling of $\Delta T = 135$ K (a, b), $\Delta T = 220$ K (c, d) and $\Delta T = 265$ K (e, f).
Figures 2c, d document the details from the microstructure of the sample solidified with the initial undercooling of 220 K. In this case, austenitic grains are surrounded by carbides occurring in two morphologically different forms – carbides of $\text{M}_7\text{C}_3$ representing one constituent of eutectic colonies and skeleton shaped intergranular $\text{M}_4\text{C}_3$ carbides. In difference from previous two samples in which carbide phases of solidification origin were localised only in interdendritic and/or intercellular regions of metastable austenite, in the sample solidified from the undercooling of 265 K, the carbide phases of MC type are situated also in the volume of austenitic grains (figures 2e, f), mainly in central parts of cells and dendritic fragments. The carbide phase of $\text{M}_7\text{C}_3$ is preferably located in intercellular regions forming one constituent of two-phase eutectic. As it follows from figure 2, size and particularly morphology and location of carbide phases of solidification origin is influenced by the level of melt undercooling prior to spontaneous solidification.

3.2. Microstructures in samples after substrate solidification

Samples solidified on a copper substrate were prepared by the defined undercooling from the range of 100 K to 250 K [6]. In comparison with microstructures in samples spontaneously solidified in levitation, microstructures in splats after substrate solidification were not uniform. For example, three main zones with different microstructures were observed in the sample dropped to a copper substrate with the initial undercooling of 100 K (figure 3).

![Figure 3. Shape (a) and microstructure (b) in the sample dropped to the copper substrate with the initial undercooling of 100 K.](image)

![Figure 4. Details from the microstructure of the sample solidified on a copper substrate with the initial undercooling of 100 K in the bottom part (a, b), in the middle part (c, d) and in the top part (e, f).](image)
In the very narrow zone solidified near the substrate, fine columnar grains of solid solution (austenite) and $M_7C_3$ and $M_4C_3$ carbides with various morphologies are present (figure 4a). $M_4C_3$ carbide phases are preferably located in the growth direction of columnar grains (figure 4b). Locally situated $M_6C_3$ carbides (eutectics $M_4C_3 +$ austenite) of sponge shape can be observed inside of columnar austenitic grains. In the middle parts of a splat, dendritic and cellular forms of austenite were identified. The volume of austenite, no carbides are visible. The detail from microstructure after deep etching in figure 4d documents the different morphology of $M_7C_3$ and $M_4C_3$ eutectic carbides. The microstructure in the top parts of the splat is formed by cellular and quasi-cellular structure of austenite with the eutectic colonies ($M_7C_3 +$ austenite) in the intercellular regions and $M_4C_3$ carbides inside austenitic cells and as well as in the surroundings and volumes of eutectics on the base of $M_7C_3$ carbides (figure 4e). Continual carbide skeletons of eutectic $M_7C_3$ carbides in intercellular region and small particles of $M_4C_3$ carbides are illustrated in figure 4f. Moreover, the influence of lateral melt flow during solidification on the microstructure development can be seen from figure 3b particularly in the middle part of the splat.

4. Conclusions

Microstructures developed in undercooled samples from Cr-Mo-V hypoeutectic tool steel during spontaneous solidification in levitation and after solidification on a copper substrate were compared. During performed levitation experiments, maximum melt undercooling at the level of 290 °C below the liquidus temperature was achieved. In the samples spontaneously solidified in levitation, quasi-uniform microstructures in the whole volume of samples were observed. The main microstructural features of metastable austenite and the size, morphology and localisation of carbides of solidification origin depend on the level of undercooling reached prior to spontaneous nucleation.

In samples after substrate solidification, morphologically different microstructural regions were observed depending on the distance from the copper substrate. In the sample dropped to a substrate with the undercooling of 100 K, developed microstructures varied from the fine columnar structure very near the substrate through the dendritic/cellular in the middle parts to the (quasi)cellular at the top of a splat.

In the samples solidified in levitation and also on a substrate, the $M_7C_3$ carbide phase was located in eutectic colonies at the grain boundaries of the metastable austenite. The $M_4C_3$ carbides were situated in interdendritic and/or intercellular regions. Moreover, in the sample most undercooled prior to solidification and in the top parts of the sample solidified on a substrate, the $M_4C_3$ carbides were observed also in the volume of austenitic grains.

5. References

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