

Semi-Quantitative Model of the Microstructure Development in the High-Alloyed Iron Based Alloy During Atomization

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Received: December 8, 2011; Revised: March 15, 2012

The paper deals with the analysis of microstructure formation in the tool steel of ledeburite type Ch12MF4 with the chemical composition of 2.37% C, 12.06% Cr, 1.2% Mo and 4.0 % V [wt. (%)] in the process of nitrogen gas atomization. Three main types of solidification microstructures were observed in rapidly solidified powder particles: dendritic, compound and cellular. Based on the morphological features of microstructures observed in rapidly solidified particles and mathematical modeling of the thermal history of solidifying spherical droplets, the semi-quantitative model of the microstructure development in the Ch12MF4 steel during atomization was suggested. According to this model, it is supposed that the transition from dendritic to partially dendritic (compound) and nondendritic microstructures results from the thermally induced fragmentation of dendrites by the mechanism of their remelting, morphological changes of dendrite fragments and following spheroidization. The intensity of dendrite fragmentation in solidifying particles of different diameters is controlled mainly by the recalescence temperature and duration of quasi-isothermal period of solidification.

Keywords: *tool steel of ledeburite type, gas atomization, undercooling, rapid solidification, recalescence, dendrite fragmentation*

1. Introduction

Gas atomization of molten alloys belongs to the technologies of materials production exploiting benefits of rapid solidification processing. During gas atomization, a molten stream is disintegrated in several stages by the impact of a high velocity gas flow into quasi-spherical droplets of different sizes¹⁻². Moreover, many collisions of droplets in different phases of solidification result in formation of powder particles with satellites, splats, small penetrated particles into larger droplets, etc.

In the process of gas atomization, the droplets are cooled down by the atomizing gas. Depending on the melt purity, the cooling gas, cooling rate and other parameters, nucleation of solid phase occurs at a certain temperature below the liquidus temperature. Rapid nonequilibrium solidification of an undercooled droplet then proceeds in several stages. After nucleation, the phase of recalescence or quasi-adiabatic period of solidification occurs. During this period, the temperature of the droplet rapidly increases due to the latent heat release. In the next period of quasi-isothermal plateau, the temperature of the droplet remains almost constant as the latent heat released and the heat extracted from the droplet to the gas per unit time are approximately equal. The final stage represents the cooling of the solidified particle.

In the rapidly solidified (RS) powder particles, various types of primary solidification microstructures can be usually observed³⁻¹⁰. The development of final solidification microstructures in RS particles is determined mainly by

thermodynamic and thermokinetic conditions of rapid cooling, nucleation and solidification of the melt¹¹⁻¹³. In various metallic systems, the phenomenon of grain refinement during rapid solidification of highly undercooled melt due to the dendrites fragmentation was observed¹⁴⁻¹⁷ and theoretically explained by Karma's model^{14,18}.

To predict the conditions favourable for initiation and accomplishment of the process of microstructure transition from dendritic to cellular in the atomized powder particles, mathematical modeling and numerical analysis of the thermal history of a single particle during atomization can be used. Of course, this approach represents quite difficult task because of number of parameters and variables unknown and not measurable during atomization.

This paper deals with the analysis and explanation of microstructure development in the rapidly solidified powder particles from the hypoeutectic tool steel of ledeburite type Ch12MF4 prepared by nitrogen gas atomization. Three main types of solidification microstructures were identified in RS powder particles: dendritic, compound (a mixture of dendritic and cellular) and cellular¹⁹⁻²⁰. It is supposed that the transition from dendritic to compound and cellular microstructure occurs by the mechanism of thermally induced fragmentation of dendrites. The microstructures documented the procedure of dendrites fragmentation and microstructure refinement in Ch12MF4 alloy are presented. Mathematical model prepared and applied for the analysis of the thermal history of a single undercooled and rapidly solidified spherical droplet in the atomization process is

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briefly described. Based on the comparison of results of mathematical modeling and microstructural observations, the semi-quantitative model of the microstructure evolution in the RS powder particles of the Ch12MF4 steel during atomization is suggested. Thermophysical conditions and mechanisms of formation different types of solidification microstructures in single RS powder particle are discussed.

2. Experimental Material and Methods

The RS powder of the tool steel Ch12MF4 of ledeburite type with the chemical composition of 2.37% C, 12.06% Cr, 1.2% Mo, 4.0% V and balance Fe [wt. (%)] was produced by the nitrogen gas atomization in the industrial conditions. Mounted, polished and etched cross-sections of the RS particles were previously microscopically examined¹⁹⁻²⁰. Phase identification was performed by transmission Mössbauer spectrometry and X-ray diffraction analysis¹⁹. For the more detailed characterization of microstructural features, scanning electron microscope JEOL JSM 7600F was used.

The quasi-equilibrium solidification behavior of the Ch12MF4 steel was investigated by differential thermal analysis (DTA)¹⁹⁻²⁰. The experiments revealed that solidification of the steel starts by the crystallization of austenite at the temperature of 1324 °C. This value is in good agreement with thermodynamic calculations for this type of steel²¹. Solidification is finished by two eutectic reactions initiated at the temperatures of 1253 and 1239 °C during which two types of morphologically different carbides, MC and M₇C₃, are formed. The quasi-equilibrium solidus temperature was found to be 1225 °C.

To investigate the attainable level of undercooling in this type of steel and the influence of nucleation temperature on the microstructure development, the levitation experiments were performed²²⁻²³. The maximum undercooling at the level of 300 K was obtained in spontaneously solidified samples during levitation experiments while the average undercooling was 214 °C. The maximum of attained undercoolings in the investigated steel correspond to values reported for pure iron²⁴ and iron based alloys²⁵⁻²⁷.

Results from microstructural and phase analyses of the RS particles, DTA experiments, thermodynamic calculations and levitation experiments were used as input data for the build-up of semi-quantitative model of the microstructure development in the RS powder particles of hypoeutectic Cr-Mo-V tool steel during nitrogen gas atomization of the melt.

3. Mathematical Model

In order to analyse the material behavior during rapid solidification of discrete particles in gas atomization process and to quantify the thermal effects, physical and thermokinetic conditions governed the final microstructure development, newtonian thermokinetic mathematical model briefly described in the next section was developed.

The model supposes that a droplet created in gas atomization process keeps spherical shape, its deformation due to the surface tension and thermal expansion are neglected. Thermophysical properties of liquid and solid

phases are different and temperature dependent. The heat removal from the droplet surface to the surroundings is carried out by the mechanisms of convection and radiation while the combined heat transfer coefficient depends on the droplet/gas relative velocity and the droplet surface temperature. Generally, solidification can start by homogeneous or heterogeneous nucleation from one or multiple nucleation events. In this work, it is supposed that the nucleation occurs at the defined temperature in order to study its influence on the thermal history of RS droplets with different diameters. During solidification, the solid-liquid (S-L) interface velocity is dependent on the melt undercooling.

According to newtonian conditions, the thermal history of a spherical droplet can be described by the heat balance equation. The enthalpy decrease with the time is equal to the heat extracted per time unit from the droplet surface to the surroundings

$$-\frac{dH}{dt} = h(T - T_f)S \quad (1)$$

The heat removed through the droplet surface S depends on the combined heat transfer coefficient h and the temperature of atomizing gas T_f .

Taking into account the enthalpy change of liquid and solid phases and the latent heat released, the time variation of the droplet temperature becomes

$$\frac{dT}{dt} = \left[-\frac{6h(T - T_f)}{d} + \rho_L l_f \frac{df_S}{dt} \right] \left[\rho_S c_S f_S + \rho_L c_L (1 - f_S) \right]^{-1} \quad (2)$$

where d is the droplet diameter, ρ and c are the density and specific heat of solid (S) and liquid (L) phases, respectively, l_f is the latent heat and f_S is the solid fraction.

The combined heat transfer coefficient can be estimated using Ranz-Marshall criterial equation for computation of convective heat transfer coefficient h_K ^[28]

$$h = h_K + h_R = \frac{\lambda_f}{d} \left[2 + 0.6 \text{Re}^{1/2} \text{Pr}^{1/3} \right] + \varepsilon \sigma_0 (T^2 + T_f^2)(T + T_f) \quad (3)$$

in which $\text{Re} = \frac{v_f d}{\nu_f}$ is the Reynolds number, $\text{Pr} = \frac{\nu_f}{a_f}$ is

the Prandtl number, $v_r = |v_f - v_d|$ is the relative velocity of the gas and the droplet, ν_f is the kinematic viscosity, λ_f is the thermal conductivity, a_f is the thermal diffusivity of atomizing gas, ε is the droplet emissivity and σ_0 is the Stefan-Boltzmann constant.

The Boettinger-Coriell-Trivedi model²⁹ was applied to compute the relationship between the S-L interface velocity v_{SL} and undercooling ΔT for the solidification of austenitic dendrites in the form $v_{SL-D} = 2.668 \times 10^{-8} \Delta T^{2.995}$. To describe the solidification kinetics for the eutectic growth, the Magnin-Kurz-Trivedi for irregular eutectic growth³⁰ was adopted ($v_{SL-E} = 1.422 \times 10^{-6} \Delta T^2$).

4. Results and Discussion

In the RS powder particles from hypoeutectic tool steel Ch12MF4, three main types of solidification microstructures were found (Figure 1): dendritic, compound (a mixture of dendritic and cellular) and cellular. Dendritic microstructure represents the dominant morphological variant in the rapidly

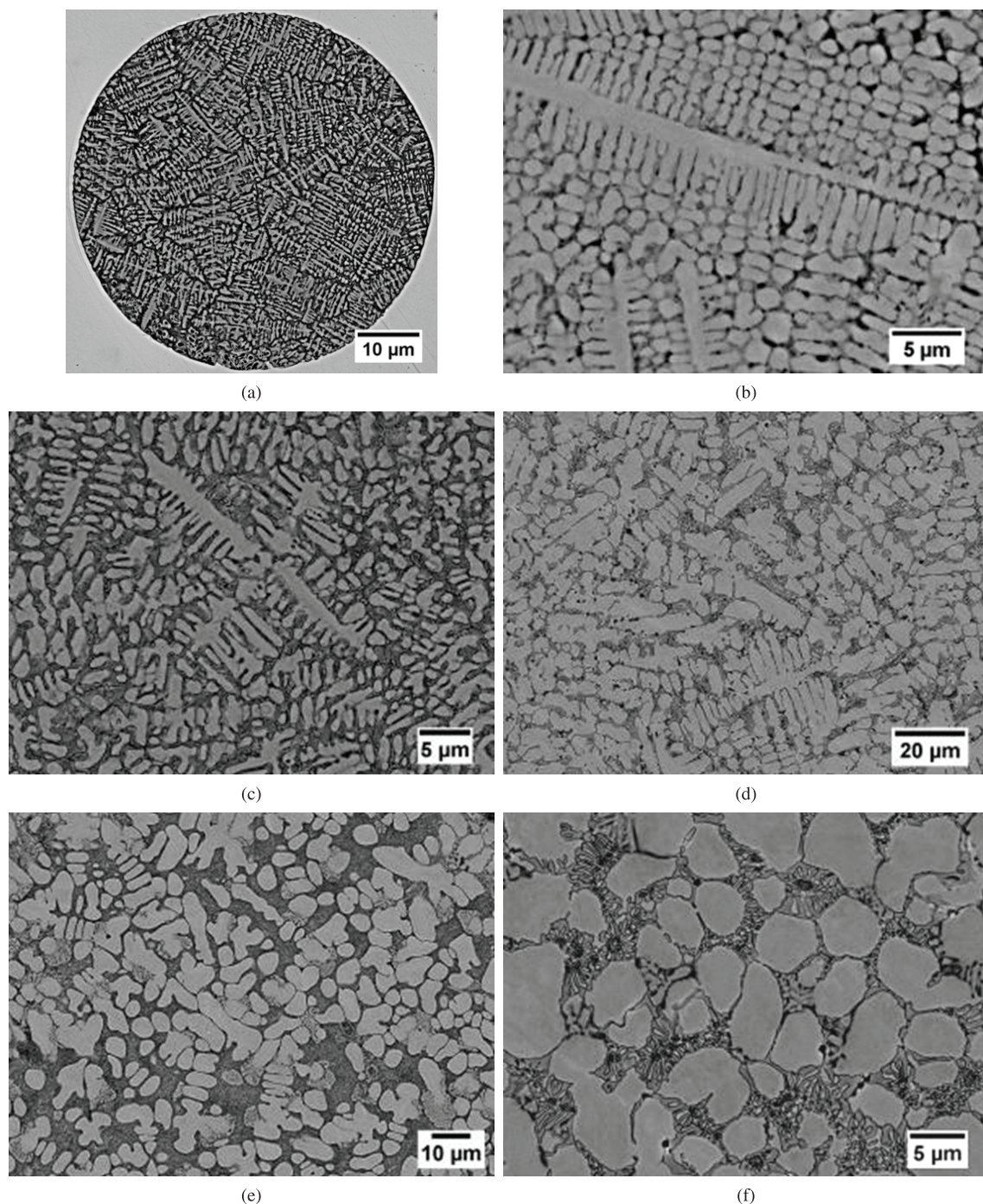


Figure 1. Microstructures in the rapidly solidified powder particles from the Ch12MF4 steel after nitrogen atomization documented the thermally induced transition from dendritic to cellular microstructures a) dendritic blocks in a RS particle, b) fragmentation of secondary dendrite arms, c) spheroidization of fragments from secondary dendrite arms and the break-up of dendrite trunks, d) the break-up of dendrite trunks and progressive spheroidization of fragments, e) advanced stage of dendrite fragmentation and spheroidization, f) cellular microstructure.

solidified particles. The percentage population of compound and cellular microstructures increases in larger powder particles²⁰. Cellular microstructures were found mainly in the particles more than 200 μm in diameter.

Dendritic microstructure is comprised of differently oriented dendritic blocks formed by dendrites of

metastable austenite and fine austenite-carbides eutectics located in the area between secondary dendrite arms (Figure 1a). The transition from dendritic to cellular microstructure starts by the separation of secondary dendrite arms from the primary arm (Figure 1b). The primary arm remains continuous, unbroken with the initial shape and size

of dendritic-carbide block. The process of microstructure transition follows by fragmentation and spheroidization of fragments from secondary dendrite arms and by the break-up of dendrite trunks (Figures 1c, d). The detail in Figure 1e illustrates the local narrowing of dendrite arms and formation of fragments and quasi-globular particles in the advanced stage of dendrite fragmentation. The cellular microstructure developed by the mechanisms of thermally induced dendrite fragmentation and spheroidization of dendritic fragments contains globular austenitic particles located in eutectic austenite-carbide mixture of phases (Figure 1f).

Figure 1 documents the progress of microstructure transition in RS powder particles from dendritic to cellular by thermally induced fragmentation of dendrites and following spheroidization of dendritic fragments. The principal assumption for the occurrence of thermally induced morphological changes of austenitic dendrites in the solidifying droplets is the heating of an undercooled droplet due to the latent heat released during the phase of recalescence above the temperatures of eutectic transformations, i.e. above the temperature of $T_{E1} = 1253\text{ °C}$. That means, that the solidifying microvolumes of the steel in which morphological transitions take place must have temperatures from the temperature interval corresponding to the stability of austenite and the melt, i.e. at the temperatures between the liquidus temperature of 1324 °C and the temperature of the onset of eutectic reactions, $T_{E1} = 1253\text{ °C}$. In addition, the time during which the solidifying droplet remains in this temperature interval should be sufficient for:

- fragmentation of secondary dendrite arms (Figure 1b);
- fragmentation of dendrite trunks (Figures 1c, d);
- spheroidization of dendrite fragments (Figures 1e, f).

If the rapidly solidifying undercooled two-phase (melt + austenite) droplet from Ch12MF4 steel during the development of austenitic dendrites in the phase of recalescence does not exceed the temperature of

1253 °C , there are not favorable conditions for the morphological changes of dendrites established and the dendritic morphology of austenite developed during rapid solidification is maintained.

Issuing from these basic thermal assumptions and taking into account the results of numerical simulations presented below, the characteristic time-temperature conditions necessary for the development of dendritic, compound and cellular microstructures in the RS powder particles from the Ch12MF4 steel can be approximately quantified.

Numerical simulation of the process of cooling and rapid solidification of spherical particles from Ch12MF4 steel during nitrogen gas atomization was performed using the described newtonian mathematical model for the droplets with diameter from 40 to 300 μm . The initial nitrogen gas velocity was supposed to be 300 and 100 m/s, respectively. The droplet originated in the process of atomization is cooled down by atomizing gas from the initial temperature of 1400 °C by the mechanisms of convection and radiation. The values of combined heat transfer coefficient depend mainly on the relative velocity of the droplet and gas. They achieve the maximum values at the beginning of the cooling process (Figure 2a) resulting in the droplet cooling rates or order from 10^4 to 10^5 K/s (Figure 2b). In order to study the influence of nucleation temperature on the thermal history of RS particles, it was supposed that solidification of molten droplet starts by nucleation from multiple solidification events at the given temperatures from the interval from 1000 to 1300 °C corresponding to the droplet undercooling under equilibrium liquidus temperature from 324 to 24 K. The assumed values of initial undercooling are comparable with undercoolings of molten samples from this type of steel attained during levitation experiments²³.

Figure 3 illustrates the thermal histories of droplets with diameter of 60 and 300 μm starting solidification at chosen nucleation temperatures T_N . The overall duration of the cooling and solidification of the larger droplet is substantially longer. The decrease of the nucleation temperature results

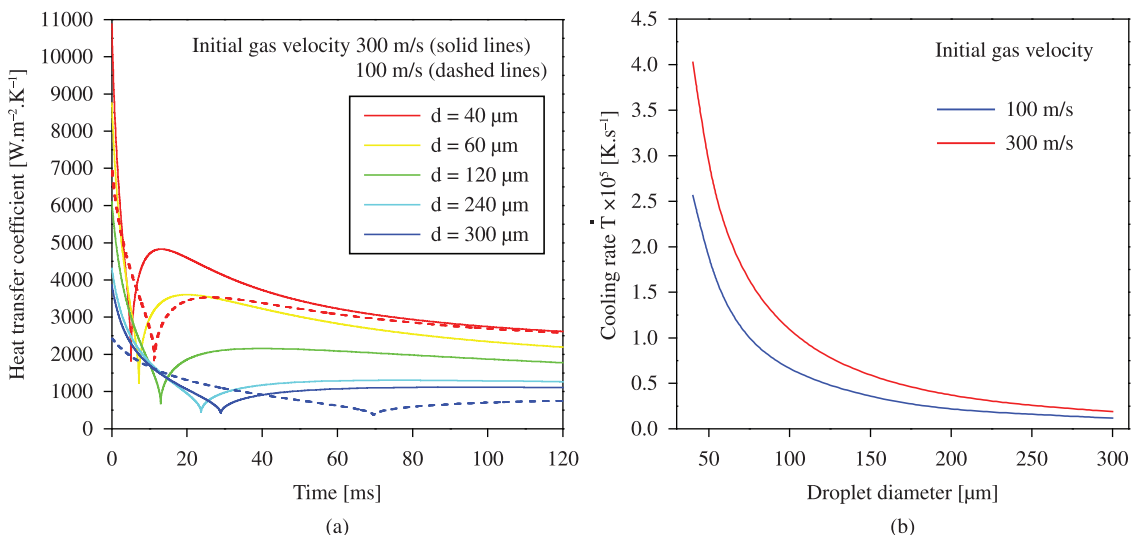


Figure 2. Cooling conditions of droplets in nitrogen gas atomization a) time dependence of the heat transfer coefficient for droplets with different diameters and b) resulting maximal cooling rates of droplets.

in the lower recalescence temperature and the shorter time of quasi-isothermal period of solidification. In the case when the solidification starts by nucleation at the highest temperature of 1300 °C, the temperature of the droplet at the beginning of the solidification decreases as the rate of latent heat released during the initial stage of solidification is smaller than the heat extracted from the droplet per unit time to the surroundings. Consequently, the phase of recalescence occurs with some delay. This effect is more significant for smaller droplets which cooling rates are higher. The decrease of initial gas velocity from 300 m/s (solid lines) to 100 m/s (dashed lines) has no relevant influence on the recalescence temperature which is governed mainly by the nucleation temperature. It results only in the extension of the duration of quasi-isothermal plateau.

As it follows from Figure 3 and Figure 4a, molten droplets started solidification with the maximum considered

undercooling at nucleation temperature of 1000 °C are heated up by the latent heat released during recalescence not even to the equilibrium solidus temperature of the investigated steel. Duration of the phase of quasi-isothermal plateau is less than 0.2 milliseconds (Figure 4b).

If the rapid solidification of droplets with diameter from 40 to 300 μm begins at the nucleation temperature of 1100 °C, the recalescence temperatures are lower than the eutectic temperature $T_{E1} = 1253$ °C. Based on these results it can be concluded that in droplets from Ch12MF4 steel deeply undercooled prior to solidification below the temperature of 1100 °C, the thermal conditions favorable for the re-melting and fragmentation of austenitic dendrites during their solidification are not generated and consequently, the final solidification microstructure is dendritic. On the other hand, when taking into account the highest assumed nucleation temperature of 1300 °C,

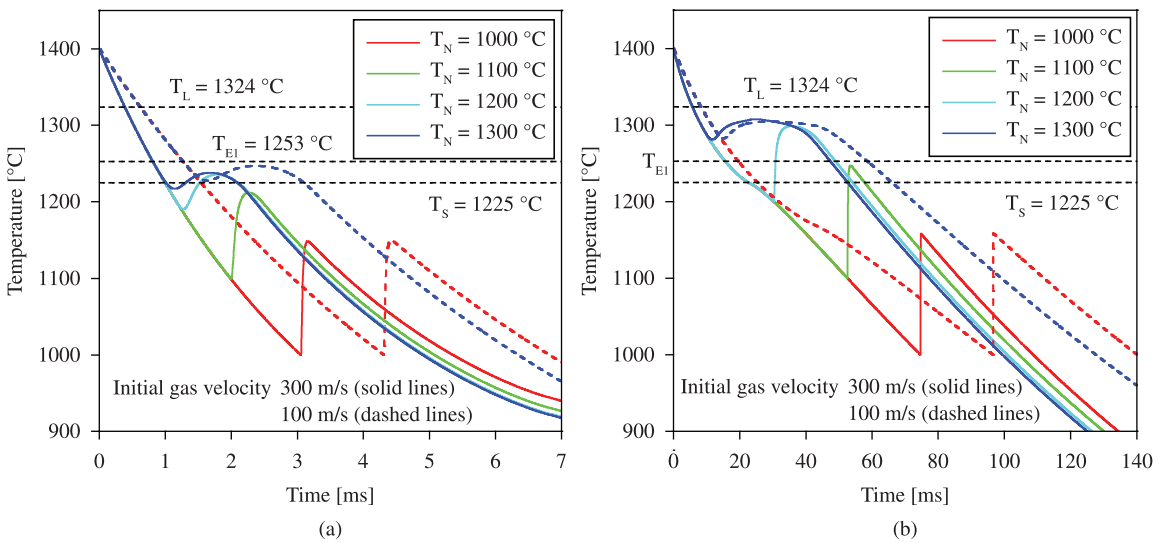


Figure 3. Thermal histories of droplets with diameter of a) 60 μm and b) 300 μm rapidly solidifying from different nucleation temperatures T_N .

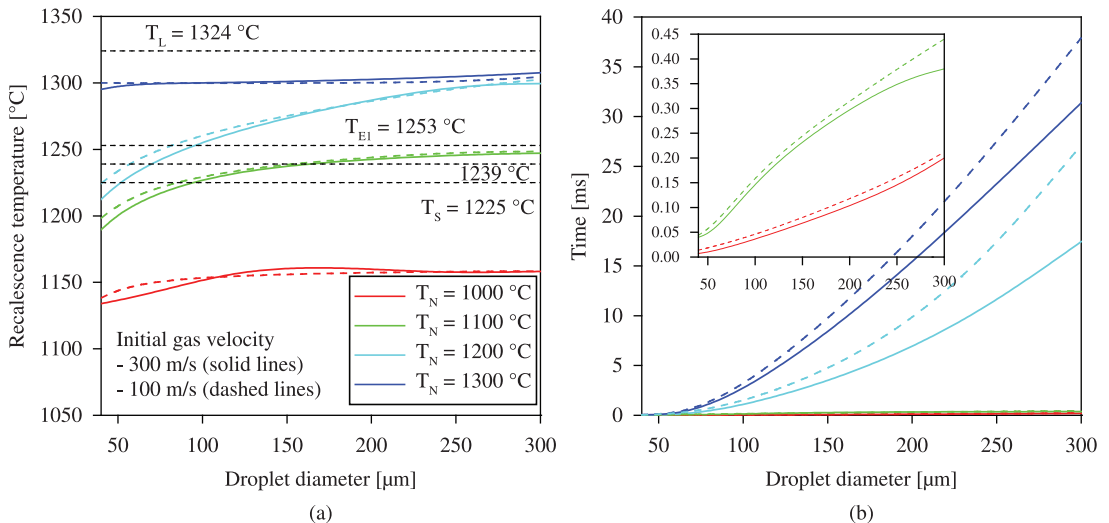


Figure 4. Dependence of a) the recalescence temperature and b) duration of quasi-isothermal plateau on the droplet diameter for different supposed nucleation temperatures T_N and initial gas velocities of 100 and 300 m/s.

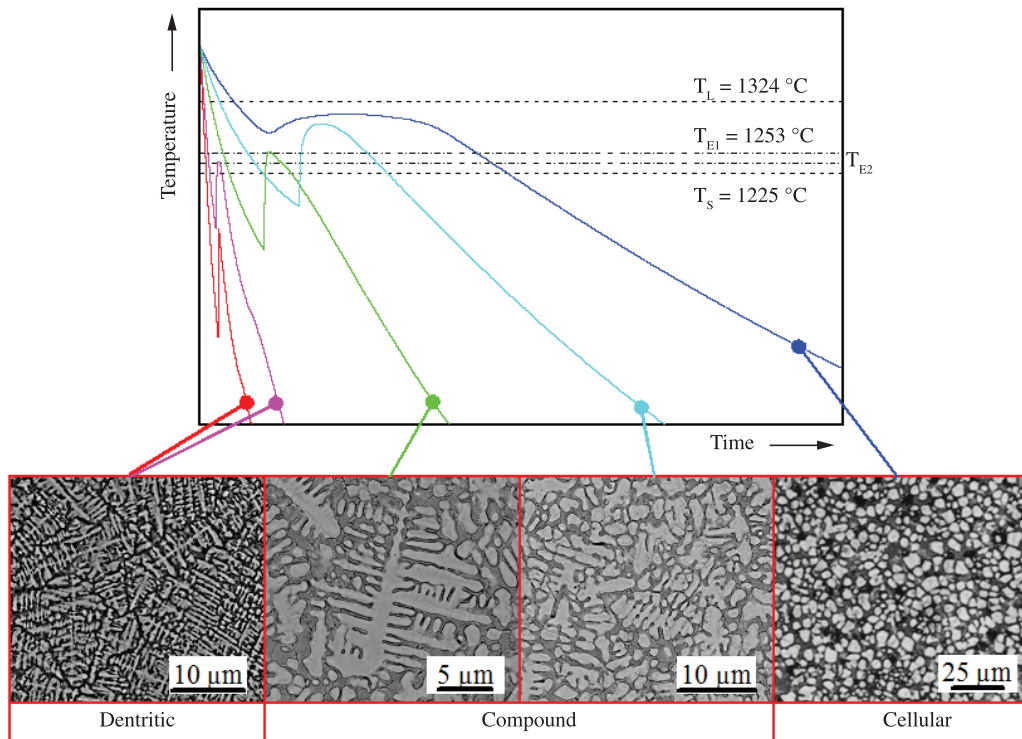


Figure 5. Semi-quantitative model of microstructure development in the rapidly solidified powder particles of Ch12MF4 steel.

the recalescence temperature is approximately 50 °C above the eutectic temperature $T_{E1} = 1253$ °C for droplets of all considered diameters even if the initial velocity of atomizing gas is lower (Figure 4a). Duration of the period of quasi-isothermal plateau for the largest droplets exceeds 30 ms. It can be supposed that these conditions are sufficient for the fragmentation of austenitic dendrites and also for the following spheroidization of dendrite fragments resulting in the cellular microstructure development.

The recalescence temperature is also higher than the eutectic temperature of 1253 °C if the supposed nucleation temperature is 1200 °C and the droplet diameter is larger than 95 μm. The time during which the droplet remains at the post-recalescence temperature is more than 1 ms. For droplets larger than 95 μm in diameter, the recalescence temperature increases up to approximately 1300 °C for the droplet with diameter of 300 μm while the time of quasi-isothermal plateau is more than 15 ms taking into account the initial nitrogen gas velocity of 300 m/s. It can be supposed that in the droplet with diameter larger than 95 μm solidifying from the nucleation temperature of 1200 °C, the thermally induced transformation of microstructure can occur. The level of fragmentation of austenitic dendrites and spheroidization of dendrite fragments depends on the duration of the period of quasi-isothermal plateau.

Based on the presented computation results and detailed microstructural analysis, the semi-quantitative model for the microstructure development in the rapidly solidified powder particles of Ch12MF4 steel was developed (Figure 5). The model involves calculated thermal histories of droplets and

corresponding characteristic microstructures taking into account the temperatures of phase transformations measured by DTA and verified using thermodynamic calculations. If the temperature of the droplet during solidification is lower than the equilibrium solidus temperature or the temperature of the eutectic reaction $T_{E1} = 1253$ °C, the dendrites of metastable austenite developed in the phase of recalescence remain unchanged.

If the recalescence temperature is higher than both eutectic temperatures, thermally induced transition of austenitic dendrites can occur resulting in compound or cellular microstructure development. The level of fragmentation of dendrites and spheroidization of dendrite fragments depends on the recalescence temperature and duration of the phase of quasi-isothermal plateau. In the particles heated during recalescence to the highest temperatures near the equilibrium liquidus temperature and remaining at this temperature during sufficiently long time, the cellular microstructure is formed.

5. Conclusions

Three main types of solidification microstructures were identified in the rapidly solidified powder particles from the Ch12MF4 tool steel of ledeburite type prepared by nitrogen gas atomization – dendritic, compound and cellular. The typical microstructures, progress of morphological changes and microstructure transition from dendritic to cellular were documented. Using developed mathematical model, the influence of the nucleation temperature on the thermal history, recalescence temperature and duration of

quasi-isothermal plateau for the particles with diameter from 40 to 300 μm solidifying during gas atomization with the initial nitrogen gas velocity of 100 and 300 m/s was investigated and discussed. Based on the comparison of results of mathematical modeling and microstructural analyses, the semi-quantitative model of the microstructure evolution in the RS powder particles from the Ch12MF4 steel during atomization was suggested. The model illustrates the typical time-temperature conditions favorable for the development of main types of microstructures and transition from dendritic to cellular microstructure

in the powder particles from investigated steel due to the thermally induced fragmentation of austenitic dendrites and spheroidization of dendrite fragments.

Acknowledgements

The research has been supported by the Scientific Grant Agency of the Slovak Republic (VEGA) within the project n° 1/1041/11 and the project ITMS 26220120048 under the Research & Development Operational Programme funded by the ERDF. The authors are particularly grateful to L. Čaplovič for valuable discussion.

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