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Nanodispersing and Hardening in Technology of Selective Laser Melting of Metallic Powders

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Abstract. The structure of materials produced by selective laser melting from metal powders of different chemical composition is analyzed. For all samples obtained by melting, studies showed an increase in the strength characteristics over similar indices of monolithic samples of identical chemical composition. In the set experiments, the effects of the anisotropy of the properties, due to the samples growth direction, and hardening, which linearly increased with increasing of the laser energy, were recorded. A phenomenological model of phase nuclei formation is formulated by the mechanism of condensation of metal vapors formed above the melting zone. Completeness of the crystallization process determines the quality of the material obtained. It has been established that nanoparticles of vapor condensation above the melting zone and the peculiarity of the structure of materials obtained by selective laser melting, in the form of a combination of ultradispersed particles of a condensed state and needle-like crystallites (ultrafine-grained structure of crystallization) are factors of strength enhancement. The ultra-fine-grained structure of the needle-shaped species is formed by the mechanism of epitaxy at the stage of crystallization in microvolumes with a melt, depending on the temperature conditions.

1. Introduction

Manufacturing of complex shape digitally designed products by selective laser melting (SLM) of layerwise raw powder has a great practical and scientific interest [1-4]. Among SLM's special features there are solid and liquid phases and their interaction when shortly exposed to a laser beam. Local melting and structural-phase transformations occurred in thermodynamic nonequilibrium have not been sufficiently studied, although they are widely used in different technological applications with transient modes.

The paper's objective is to summarize the available research on metal design structures of different chemical composition manufactured by SLM and to indicate factors adding to the strength increase of manufactured material.

2. Materials and experimental methods

SLM of powders has been performed on EOSINT M270 with 200W solid-state laser that continuously scans the powder melting zone at up to 800 mm/s. (Fig. 1). Thermal energy of the laser is sufficient to melt the powder particles of up to 40 μm .



The experiments have involved powders with chemical composition of general stainless steel grades (Table 1). The powder of unalloyed technical iron has also been used to eliminate the influence of chemical composition on the strength of material properties.

To evaluate mechanical properties we have grown samples for standard trials by dynamic bend (ASTM E8 and KCU) and uniaxial tension (ASTM D 6110).

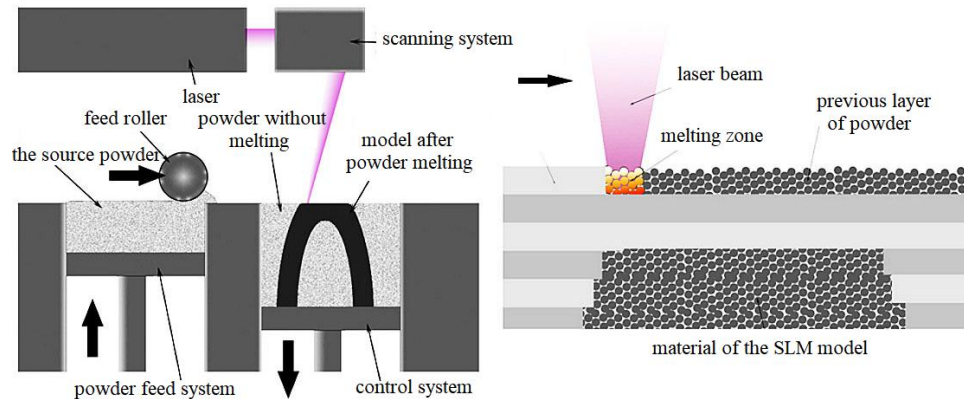


Figure 1. Scheme of selective laser melting process.

Table 1. Chemical composition of powders.

Steel grades	Weight of basic alloying elements (%)						
	C	Si	Mn	Cr	Ni	Mo	Ti
17-4PH	0,07	0,38	0,7	15,7	4,0	—	0,1
316L	0,03	—	1,5	16,8	13,3	2,6	—
321	0,12	0,8	0,8	17,2	10,6	—	0,8
Fe	0,019±0,001	0,07±0,04	0,08±0,06	—	—	—	—

Structural studies have been performed by Axio Observer light microscope and FEI Tecnai electron scanning microscope. Image processing has been performed by digital metallography, including multifractal analysis to detect structural features caused by melting and solidification transience in inhomogeneous temperature fields. Multifractal assessment of halftone images of structures has been performed by their black-and-white (binary) mappings, which are mathematically considered as statistical sets of different dimensions [5]. The required parameters have been calculated from the spectrum of dimensions D_q : the spatial dimension D_0 , the ordering measure $\delta = D_1 - D_q$ and the periodic measure $K = D_{-q} - D_{+q}$. Q refers to dimensions of the monofractal statistical binary subsets in the interval $(-40 \div +40)$ that resulted in the binary map of the original halftone image.

The spatial dimension D_0 depends on dimensions of the structural changes zone, and it is a quantitative estimate of the image characterizing filling of the image field. The δ value is affected by symmetry breaking for the overall configuration of the whole structure. The larger δ (in absolute value) the more structural order, while tent of δ to zero is a sign of chaos. An increase of K indicates an increase of periodic component in inspected mapping of the structures. The results of multifractal assessment have a probability statistical sense [6], and the use of analysis is determined by physical content of the problem.

3. Experimental results and discussion

According to the mechanical testing results, the strength parameters (hardness, tensile strength, yield strength) of the samples manufactured by SLM have been repeatedly exceeded by the similar indicators of solid samples with identical chemical composition [7,8]. The experiments have revealed the effects of properties anisotropy caused by powder laying methods and hardening that linearly

increased along with the laser power growth. The features of material properties have revealed density and pores, inhomogeneities of chemical elements distribution, thermally activated diffusion processes and kinetics of structural transformations. Based on the phase transformation models in steels review [9], the growth of strength properties could presumably be caused by the interaction of atoms in the zones of melting and solidification of contacting particles. Therefore, the alloyed compositions were expected to have clusters of complex elements including Fe, Cr, Ni [10] that in case of rapid cooling would become centers of crystallization and phase formation. Electron microscopic examination of the fracture surfaces and metallographic sections of the testing samples of stainless steels did not contradict the scheme described above. But the testing of the powder samples of technical iron, manufactured by SLM, required additional check and analysis of the technological operations [11].

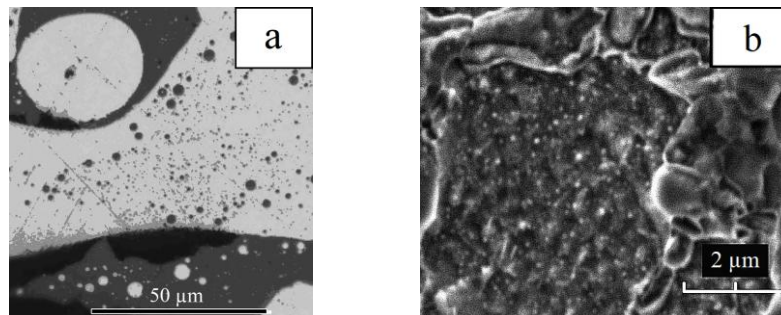


Figure 2. Spherical shape particles ranging from tenths to dozens of micrometers in microscopes: a) – light, b) – electronic bitmapped.

The light and electron bitmapped microscopy has determined that iron samples consist of spherical shape particles ranging from tenths to dozens of micrometers (Fig. 2). The regular geometric shape of particles has indicated that they were formed in a "free" (suspended) state without contacting other objects. Such phenomenon is possible if condensation of vapor (v) into a liquid (l) occurs at temperatures below $\frac{2}{3}T_m$ ¹ [12]. Being rapidly cooled, the liquid can remain in a supercooled amorphous state or crystallize (c). The metal condensation, according to desublimation mechanism $v \rightarrow c$ or $v \rightarrow l$, is subject to cooling conditions. Depending on the temperature and the elasticity of vapors, both mechanisms (heterogeneous condensation effect) can happen. During the melting of all the powders both the authors and the researchers [13] could observe traces of drop formation as luminous tracks of flying particles (Fig. 3).

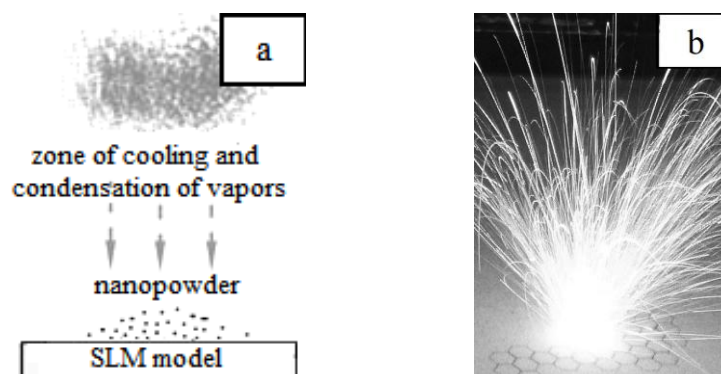


Figure 3. Settling of condensed particles on installation platform, b) – tracks of particles scattering from powder melting spot.

¹ T_m – melting temperature of metal

During the fan-shaped scattering, the hardened particles hit different parts of the powder layer. They melted where the particles were heated by laser. There where the laser beam no longer had a thermal effect, the particles remained more or less crystallized. It can be assumed that the remaining small-sized particles acted as mechanisms both hardened by precipitates and activation of epitaxy processes in micro melting with a new layer of powder. Besides, apart from the chemical composition of powder, the temperature in laser spot and the cooling conditions of reaction, the powder layer thickness significantly affects kinetics of phase transformations and amorphous into crystal transition. A wide range of conditions from nonequilibrium to almost equilibrium determines a wide range of material properties and conditions of directional adding (epitaxy) of powder layers.

Analyzing the images of structures, we have noticed that the smallest particle size formed in the reactions $v \rightarrow l$ or $v \rightarrow c$ made $0,15 \pm 0,05 \mu\text{m}$. The similar in size regular shape precipitates have been revealed in the images of steel structures of different chemical composition manufactured by SLS and been observed by SEM and TEM methods in 316L steel [14,15]. Consequently, this technological effect commonly occurred in SLM process, and it is subject to be analyzed individually.

Physical thermofluctuation theory of strength [16] says that fracture events followed by nucleus microcracks formation of $0,15 \pm 0,05 \mu\text{m}$ are caused by sublimation process due to irreversible atomic bond ruptures. The observed opposite process of steam consolidation into spherical particles of $0,15 \pm 0,05 \mu\text{m}$ results from physical objects formation process by means of atomic bonds order depending on the atoms coordination environment. About 10^7 iron atoms with atomic distance of approx. $0,3 \text{ nm}$ can take part in minimum spherical particles formation that topologically is enough for such reaction. With rapid cooling and vapor cloud compression, the efficiency of expected scenario is determined by vapor density fluctuations together with prevalent contributions of recurrent component in interaction potential. Since the final stage of atomic bonds formation goes along with its volume decrease, the process can be treated as a first-order phase transition.

Implemented in non-equilibrium thermodynamic conditions of liquid-solid interaction, condensed spherical particles are the basis of new objects formation like grains and crystals.

During crystallization their morphology and growth direction, stability of the crystal lattice, relaxation potential by shifts and structural order are determined by temperature fluctuations and chemical composition in the melting zone, optional convection, impurities and other factors. Although there is no true connection evidence between the internal structure of materials and the multifractal parameters of structures mapping, processing of halftone grain images by statistical-geometric approximation provides additional numerical data. Images of similar size grains disclosed by chemical etching on stainless steel section (Fig. 4, 5) have been chosen as an example of multifractal parametrization of structures. The grain images are processed under the same procedure.

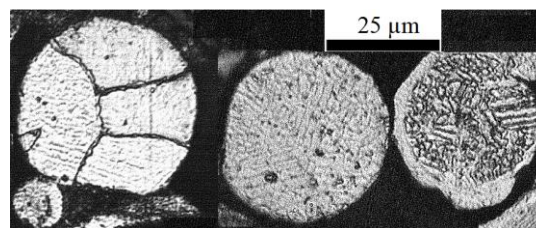


Figure 4. Similar size grains of different structure, detected on a 321 stainless steel section.

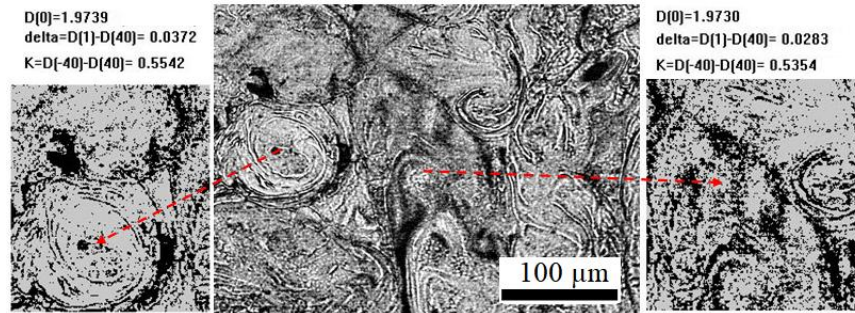


Figure 5. Multifractal certification of 321 steel structure.

The multifractal parametrization of images of steel structures manufactured by SLM has shown (Fig. 6) that, despite the short exposure periods in laser beam, the regular optional structure typical for crystallization dominates in solid and liquid metal. However, D_0 , δ and K value spreads mean that grains and crystals are formed in different conditions and characterized by different morphologies of structures. For example, the grains can have signs of disordered or amorphous state, pores (small D_0 and δ) and the ordering behavior (growing δ) with simultaneous increase in periodicity (K scatter decrease) and porosity decrease (growing D_0).

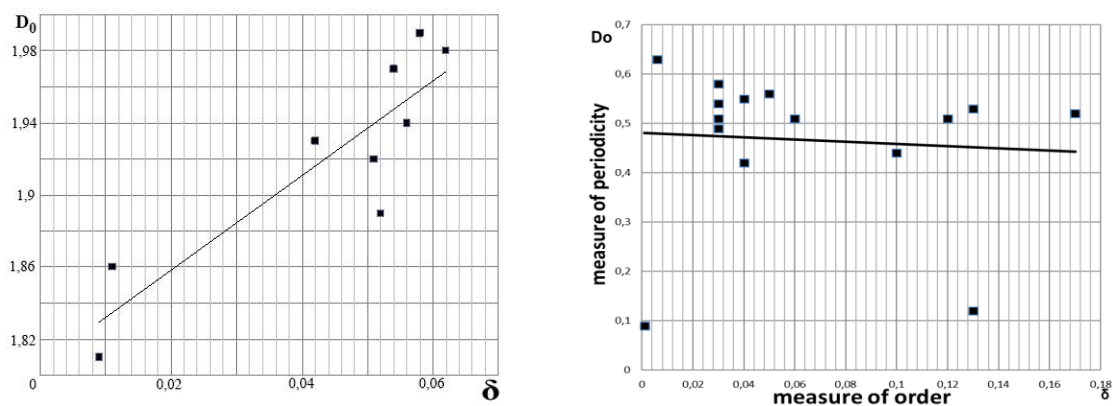


Figure 6. Results of multifractal certification of grain images, shown on Fig. 4.

In spite of the fact that the multifractal parametrization data of binary maps are not identified with real structures, we have reasonably used the results of multifractal image validation of structures. We also assume that the data albeit indirect, provide additional information on the structure of the observed objects.

4. Conclusions

1. When producing materials by selective laser melting, the nuclei of a new structure or phase are formed not in volumes between liquid and solid phases, but done rather earlier over the melting spot in vapor by interatomic interaction within desublimation or crystallization transition from vapor into solid.
2. The minimum condensed particle size as nucleus of a new structural state (phases) is $0,15 \pm 0,05 \mu\text{m}$.
3. Liquid and solid interaction of condensed particles is the basis of new objects formation like grains and crystals with morphology characterized by temperature fluctuations, chemical composition in the melting zone, possible convection in the melt, impurities and other factors.

4. Structure of materials manufactured by selective laser melting as a combination of ultradispersed condensed particles and needle-shape (banded) crystallites is a factor of hardening.

5. References

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