Selective Laser Sintering of Single-Phase Powder Cr-V Tool Steel

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Presented is positive experience from selective laser sintering (SLS) of cylindrical steel specimens (3.0% C, 3.0% Cr, 1.0% Si, 12.0% V, Fe balance) 30 mm long and 5 mm in diameter by rapid prototyping. It was demonstrated that monolithic steel material could be successfully fabricated by this technology. Differential thermal analysis (DTA), scanning electron microscopy (SEM), and x-ray diffractometry (XRD) were used to study the microstructure, phase, and chemical composition of the source material and obtained specimens. Low-melting cementite-based eutectic was found to provide the liquid phase sintering of powder tool steel. The porosity of the green sintered specimens did not exceed 5%. The mean hardness value of sintered specimens was 825 HV.

Keywords powder tool steel, rapid prototyping, selective laser sintering

1. Introduction

Selective laser sintering (SLS), a form of solid freeform fabrication (SFF) technique, has recently been developed to overcome difficulties in tooling complex-shaped ceramic and metallic parts and in the long production time in fabricating prototypes.^[1] SLS employs a focused laser beam to selectively scan the powder bed surface and bind the loose powder.^[2] Numerous constraints dictate which powdered materials may be sintered. Because the laser processing occurs in a very short time, only liquid phase sintering can be realized. The liquid phase can arise because of powdered particles melting partially or throughout.

The two-component powder approach to SLS, which involves binding high-temperature ceramics such as alumina and silicon carbide with a low-melting inorganic binder, is a promising technology to fabricate ceramic composite parts.^[3-4]

The sintering of two-component powder composition is easier because one component has a lower melting point. The positive experience of direct fabrication of steel component by rapid prototyping technology is not sufficient.^[1,2] High carbon tool steels have in their structure a large amount of low-melting carbide eutectic. The present work aims to provide sintering of powder with particles having the same composition.

2. Experimental Procedure

Steel powder (3.0% C, 3.0% Cr, 1.0% Si, 12.0% V, Fe balance) was prepared by the scientific-industrial association, NPO Tulachermet. The granulometric composition of the powder and the microstructure of freshly polished laps were investigated by scanning electron microscopy (SEM) using the JSM-U3 (JEOL, Japan) electron microscope provided with a solid pair detector (SPD). Chemical composition was studied by x-ray microanalysis using a double-crystal long-wave spectrometer with a LiF and Myristat (MYR) crystal analyzer.

Temperature of phase transformations in the powder was precisely determined (approximately to 0.1°) by the microdifferential thermal analysis (DTA), using a MICRO ATD M4 apparatus (Setaram, France). Specimens (15 mg each) were heated at the rate of 10 °C/min from room temperature to 1500 °C in Ar atmosphere. Precision of temperature detection was



Fig. 1 Granulometric composition of steel powder (3.0% C, 3.0% Cr, 1.0% Si, 12.0% V, Fe balance) (**a**) ×400 (**b**) and its SEM image

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x100



x500



x1000

Fig. 2 Microstructure of the steel (3.0% C, 3.0% Cr, 1.0% Si, 12.0% V, the rest being Fe) after SLS (secondary electron image)

0.1 °C, and 1% deviation in the replicability of the start point of melting and phase transformation was obtained.

Phase composition of steel was studied using the DRON-3 x-ray diffractometer (Burevestnik, St. Petersburg, Russia), equipped with a Cr K α source and V-filter.

The following method of selective laser sintering was used. Sintering was carried out with a continuous laser LTN-103 ($\lambda = 1.6 \mu m$) in Ar atmosphere. The 5 mm laser beam was



Fig. 3 Microstructure of the steel (3.0% C, 3.0% Cr, 1.0% Si, 12.0% V, Fe balance) after SLS (SPD detector)

targeted perpendicular to the surface of powder layer. The powder was advanced onto the horizontal carrier through a slot in a lower part of the traveling bin. The layer thickness was regulated by the clearance between the bin and the surface of a preceding layer. Low-intensity vibrations generated by an electromechanical source facilitated the outflow of powder from the bin. Once the powder layer became sintered, the carrier was lowered by 700 μ m and the next layer was applied over the preceding one. The layer thickness was 700 μ m. Such SLS technology made it possible to obtain cylindrical specimens 30 mm long and 5 mm in diameter. After a 10 s sintering, the laser was turned off and a new layer was applied during 5s. Power density of the laser was approximately 200 W/cm².

3. Results and Discussion

The histograms of particle sizes and microrelief of the powders are presented in Fig. 1(a) and (b). The average size of steel particles was about 250 μ m. The temperature of common phase transformations was determined by differential thermal analysis of steel samples. Exothermic transformation with the extremum at 649 °C corresponded to decomposition of the residual austenite. The process of austenitization took place at 770-840 °C. Eutectic transformation was observed at 1135 °C. When heated above 1135 °C, the quantity of liquid phase increased, and at 1375 °C the melting of 3C-3Cr-12V steel powder was completed.



Fig. 4 Microstructure (a) and intensity distribution (b) of Ka emission for C, Cr, Si, and V



Fig. 5 X-ray diffractogram of the SLS-steel (Cr Kα emission)

Figure 2 shows the microstructure of a freshly polished steel lap after laser sintering. A good liquid phase sintering of large and small powder particles is observed. With the selected method of laser application, there develops a sufficient amount of liquid phase. This amount suffices to fill all internal cavities and to develop the dense conglomerate despite the fact that, in this experiment, the powder was not pressed, but freely poured. Each large particle of powder has a shrinkage cavity, a micropore. As the liquid phase solidifies, micropores are formed at boundaries of powder particles (Fig. 2b, c). Overall porosity of the sintered specimen is about 5% of the volume.

Microstructure was investigated by the SPD detector, which made it possible to characterize the distribution of light and heavy elements from the difference of cross sections of backscattered electrons. The image contrast in microstructure is closely related to the concentrations of elements in microvolumes. A light contrast is due to heavy elements and a dark contrast results from light elements. Figure 3 presents the microstructure of the sintered specimen obtained with the aid of an SPD detector. Lighter regions are enriched with heavier



Fig. 6 Fractogram of the sintered sample (SEM, ×5000)

components. In this particular alloy (C, Si, V, Cr, Fe) the heaviest element is Fe. Obviously, the eutectic (ledeburite) with a large volume proportion of cementite is situated along the boundaries of particles (see L on Fig. 3a). Powder particles have the disperse structure of ledeburite with patches of martensite and residual austenite in individual grains (Fig. 3b).

Figure 4(a) shows a structure of a nonetched polished lap of the laser-sintered steel. Figure 4(b) shows data of x-ray microanalysis on intensity distribution of C, Si, Cr, and V K α emissions, drawn along I-I direction in Fig. 4a.

The phase along the boundaries of powder particles is enriched with C and Si. These elements are contained in cementite Fe₃C. Vanadium carbides are distributed in the volume of powder particles. Obviously, fluctuating intensities of characteristic emissions for V and Cr along I-I direction are related to the change of their content in carbides, martensite, and residual austenite. In accordance with x-ray diffractometry (XRD), about 15% of carbide phase VC and 16% of residual austenite are present in the volume of powder particles (Fig. 5).

Hardness (HV) at 10 kg loading of steel specimens was determined for polished laps as the average for 20 measurements. At the sintered state, that was about 825 HV. Obviously, such hardness is due to phase composition of the steel containing 16% of residual austenite.

Fractography analysis was carried out to study the resistance of the sintered steel to fracture. For this purpose, cylindrical specimens 30 mm long and 5 mm diameter were impactbroken at the room temperature. Fracture surfaces were investigated by SEM.

Figure 6 shows a typical view of the steel fracture surface. The trans-crystalline fracture, which includes carbides VC, takes the whole sintered area. In the places of poor sintering, the surface of powder particles is smelted out. This can be seen from a comparison of powder particle surfaces of the starting material and that after laser action (Fig. 1 and 6).

The analysis showed that mechanical strength of the metallic body increases in microvolumes where eutectic binder content increases.

4. Conclusions

- Liquid-phase laser sintering is suitable for the monocomponent tool steel powder (3.0% C, 3.0% Cr, 1.0% Si, 12.0% V, Fe balance).
- · Optimization of parameters for laser sintering makes it

possible to obtain specimens of high density and small size (5 mm diameter and 30 mm long).

 Sintered specimens have the microstructure of ledeburite and martensite with 15% VC and 16% residual austenite. The hardness is about 825 HV.

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