Evolution of porosity depending on SLM mode and subsequent HIP processing

A A Voznesenskaya, A V Zhdanov, A S Raznoschikov

Vladimir State University named after Alexander Grigorievich and Nikolai Grigorievich Stoletovs (VlSU), 87 Gorky street, Vladimir, Russia

2obk@bk.ru

Abstract. In this work, we obtained the results of studying the effect of pore formation on the energy density deposited in the SLM process, as well as the effect of post-processing on the change in the nature of the pores. To study the porosity formed during the manufacture of parts by the SLM method, groups of primitive specimens were prepared and subjected to HIP processing. Pore formation was investigated by two methods: by optical microscopy and by weighing samples with subsequent calculations of density and porosity values. During the study of the initial samples, 4 types of pores were identified. Pore formation was investigated before and after HIP treatment. The dependence of pore formation on energy density is obtained.

1. Introduction

One of the promising technologies for additive manufacturing is the selective laser melting (SLM) technology. This method is suitable for the manufacture of parts in various industries. The part can be grown in any configuration, which is impossible with conventional machining methods. The actual direction of development of SLM can be noted in the field of production of medical equipment, cardiology and transplantology, which opens up prospects for the creation of constructively complex technical devices for medical purposes. SLM allows you to select a combination of materials that have not only the necessary physicochemical properties, excluding or reducing the likelihood of rejection by the body, but also mechanical and technological, with high hemodynamic and functional characteristics. SLM enables complex three-dimensional lattice structures to be produced [1] and also reduces production time and costs. This approach also allows the production of elements with the optimal parameters of size and performance for a particular patient without re-equipping the production line of the enterprise. Through the use of "bionic design", a compact functional execution of an individual life support system, it makes it possible not to detach the patient from everyday life, allowing him to remain a full-fledged member of society.

In selective laser melting, such phenomena as laser absorption and scattering, heat transfer, phase transformation, chemical reactions, hydrodynamics, as well as material evaporation and radiation occur [2]. One of the most common defects in SLM parts is porosity. There is a large number of works [3-7] showing the influence of certain growth parameters on the porosity of the product. Pore formation depends not only on the parameters of the melting regime, but also on the material, as mentioned in [8]. These defects can be controlled by changing the grain size and chemical composition of the starting material.

Porosity can also be eliminated by post-processing, namely by hot isostatic pressing (HIP) [9, 10]. Along with the positive research results, some works consider the negative result from the use of HIP
processing. When assessing the feasibility of using this method, it should be borne in mind that not all types of pores can be eliminated using the HIP. Gas pores located in the volume of the part and extending to the outer surfaces, the so-called "open pores", are the least affected by the HIP. The authors of [11] applied an approach to reducing porosity during subsequent HIP processing to SLM parts. Although their procedure closed the pores, resulting in a quantitatively lower porosity content. HIP is not recommended from a structural point of view, as HIP treatment can close the pores while retaining the weakness that occurs as soon as the part is exposed to high temperature or stress causing the parts to fail. For example, the authors of [12] observed that the pores that closed during HIP treatment not only reopened, but actually became larger compared to their original size before HIP. In addition, HIP can cause surface oxidation due to high temperature and pressure. Due to controversial opinions, additional studies of the nature and behavior of pores in samples obtained by the method of selective laser melting and subsequent HIP processing may still be relevant.

2. Experimental part
In this experiment, we used a powder material of the PR-12X18H10T brand with an average particle size of 40 μm (Figure 1). The shape of the particles is predominantly spherical, occasionally there are adhesions in the form of fine fractions, which will significantly affect the SLM process.

![Figure 1. SEM image of the powder material.](image)

The size of the fraction is very important in assessing the porosity of samples prepared by selective laser melting [13]. The powder must have a spherical morphology to meet the need for consistent application of uniform powder layers. Particle morphology is important for increasing the fluidity and packing density [14], [15]. Air gaps in the original powder significantly suppress compaction [16], [17]. They can be either on the surface due to moisture, which promotes the formation of gas porosity [18], or internal gas pores, which are trapped during the production of the powder. Before preparing the samples, preliminary drying was carried out to improve the melting process.

To study the porosity formed during the manufacture of parts by the SLM method, groups of samples of a primitive geometric shape were made on the industrial equipment ConceptLaser M2 Cusing. This equipment is equipped with a diode pumped fiber laser with a maximum power of 400W. The powder layer was exposed to continuous laser action: the radiation wavelength was 1070 nm, the diameter of the laser beam on the treated surface was 50 μm. In each series of samples, the parameters of the scanning speed of the laser beam were changed from 1000 mm / s to 3500 mm / s and the laser power from 150 to 350 W. In order to uniformly heat and reduce internal stresses, a standard scanning strategy was chosen: the scanning area is divided into tiny areas and processed by a laser beam in a random order. The treatment took place in an inert gas environment - nitrogen.
The resulting series of samples were subjected to subsequent heat treatment, namely HIP treatment. The process was carried out using an isostatic press, which has the ability to process samples with stepwise heating up to 2000 °C at a pressure of up to 2000 bar. This group of samples were processed at a pressure of 1800 bar in an argon atmosphere. The processing took place according to the following scheme: heating with a step of 10 °C per minute up to 980 °C, holding for 15 hours, cooling took place at a rate of 2 °C per minute up to 150 °C, then to room temperature.

3. Results and discussions

Pore formation was studied by different methods: optical microscopy (method 1) and the Archimedes method (method 2). Method 1 was carried out using an optical microscope by obtaining images in two planes: vertical and horizontal sections. The obtained images of the surface of the samples were processed in the Altami Studio program, which has the ability to investigate the geometric characteristics of objects. Analysis of micrographs is useful for assessing the size, shape and position of pores, which cannot be achieved using method 2. Among the original samples, 4 types of pores were identified, the images are shown in Figure 2.

Figure 2. Defects formed during SLM.

The first type of pores (Figure 2-1) can be designated as gas pores. Their shape is close to a circle, the sizes are comparable to powder granules (up to 40 microns). They can be characterized by gas pores. They are formed by incomplete escape of gases from the melt during laser processing. They are formed by incomplete escape of gases from the melt during laser processing. The second type (Fig. 2-2) has the form of a random nature, consisting of inscribed unsintered spherical granules, the size of such pores is from 20 microns and above. This type of pore is referred to in the literature as “keyholes” or “lack of fusion” pores, and can be found with an unmelted powder content [18]. Unmelted powder may not be present due to smudging or removal during polishing using conventional metallographic techniques. Some pores of this type are also explained by the presence of oxides in the sample, which are formed during melting and solidification [20]. The third type (Figure 2-3) has an extended shape, which consists of straight lines, angular, not rounded and does not coincide with the grinding lines, the length of the defect is comparable to that of the width, and may resemble cracks. The fourth type (Figure 2-4) of pores is crescent-shaped. The size of this type of pore is comparable to that of powder granules; the length of the arc reaches 100 μm. In works [21, 22] it is reported that the type of pore depends on the scanning speed used during processing. The higher the scanning speed, the more type 2 pores are formed, since this is due to the instability of the melt pool and incomplete melting and filling of the melt pool area.

Energy density is a defining parameter of the SLM process, which influences the formation of structure and the formation of porosity in the material. The values of porosity versus the energy input in the SLM process were obtained by calculating the ratio of the total number of image pixels to pixels corresponding to the color and brightness of the pore region. The procedure for selecting pixels that
meet the specified conditions was carried out automatically over the entire image. With this method of assessment, the measurement error is no more than 0.05 of 1%. For automated analysis, images were obtained with high contrast (to reduce the percentage of error). By adjusting the parameters for determining the range of pixels corresponding to the pores, it was possible to exclude from the calculation the defects caused by scuffing during mechanical processing of samples and the boundaries of microstructures. Figure 3 shows a plot of porosity versus energy density.

Figure 3. Graph of porosity change versus energy density.

A decrease in porosity is achieved by an increase in thermal energy in the area of melting of the granules of the powder material, which contributes to the homogenization of the melt bath, the improvement of the wettability of the powder granules and the release of gas bubbles. At a porosity of 1%, the distribution of pores is random, there are pores both at the boundary of the scanning zones of the laser beam and in the central part of the formed tracks, while the pore size is 20 - 40 μm. Initial samples with maximum porosity are formed at a flux energy density of about 10 J / mm³. The porosity decreases steadily at an energy density of 30 J / mm³. Prior to this, the porosity values vary chaotically, but in general, the porosity value does not exceed 30%. The graph shows that the HIP treatment does not always "heal the pores", in some cases the opposite effect occurs. After HIP processing, the values randomly change to a group of samples obtained at an energy density of 20%. Porosity values increase after HIP treatment in the range from 10 to 20 J / mm³. Probably, this phenomenon occurs due to visual error or due to insufficient optical power of the microscope. Micropores, which were not previously seen, probably merge into larger ones [23, 24]. Although the porosity was evaluated in two sections, the probability that this section represents the entire sample is small. This is important in the case of samples with a low percentage of pores, since the probability of missing sections with pores increases. Figure 4 shows photomicrographs of sections before and after HIP processing.
Figure 4. Micrographs of sections of SLM samples and after HIP processing

Figure 4 - A and B shows samples obtained at an energy density of 43.75 J / mm³. The "collapse" of 1, 2 and 4 types of pores is observed, while the porosity decreases by 1.5 times, which confirms the efficiency of HIP treatment for these types of pores with a porosity of less than 1%. Figure 4 - C and D shows samples obtained at an energy density of 14.29 J / mm³. Most of the pores correspond to type 2 pores, while the average porosity of the initial samples after HIP treatment increased from 5 to 15%. It can be seen that during the HIP treatment the pores changed and became much larger, the pores expanded due to other pores and were filled with powder material from the inner cavities.

A series of samples were also subjected to another assessment of porosity by Method 2, which consists in weighing and comparing the masses of equal volumes of the test substance and a liquid of known density (distilled water), called the working liquid. The density values were obtained from which the porosity was calculated (Figure 5).
Figure 5. Comparison of porosity results by two methods.

The graph shows that the porosity measured by method 2 is higher than the porosity obtained by method 1. This phenomenon is possible due to the error and inaccuracy of the Archimedes method when studying small-sized samples with low porosity. For a more accurate study, it is necessary to make a series of samples of a larger size.

4. Conclusion

In this work, we studied a series of samples obtained by the method of selective laser melting with subsequent HIP processing. 4 types of pores formed during SLM have been identified. The study of pore formation by different methods has been carried out. In the case of the study by microscopy, it was possible to obtain more accurate results than by the Archimedes method. With the help of photomicrographs, it was possible to reveal a decrease in porosity by the HIP method in samples obtained at an energy density of 20 to 50 J/mm³. At porosity values from 1 to 5%, the efficiency of HIP processing is not always positive due to the likelihood of the appearance of through or capillary pores, which can be detected by X-ray tomography. The resulting dependencies were reflected in the graphs.

Acknowledgments

This work was carried out as part of the RFBR project №20-08-00310.

References

[1] C Yan et al. 2012 Int. J. Mach. Tools Manuf. 62 32-38
[2] B Song et al 2014 Optics & Laser Technology 56 451-460
[3] J A Cherry et al 2015 The Int. J. of Adv. Manuf. Technol. 76(5-8) 869-879
[4] D Gu et al 2009 Mat. & Des. 30(8) 2903-2910
[5] L N Carter et al 2014 J. Alloys Compd. 615 338-347
[6] H Gong et al 2015 Mat. & Des. 86 545-554
[7] E Wycisk et al 2014 Phys. Procedia 56 371-378
[8] M A Melnikova et al 2017 Photonics 2 (62) 42-49
[9] R V Chkalov et al 2020 IOP Conf. Ser.: Mat. Sci. and Eng. 971(2) 022092
[10] A A Voznesenskaya et al 2020 J. Phys.: Conf. Ser. 1439 012028
[11] U Tradowsky et al 2016 Mat. & Des. 105 212-222
[12] R Cunningham et al 2017 Mat. Res. Let. 5(7) 516-525
[13] A A Voznesenskaya et al 2020 IOP Conf. Ser.: Mat. Sci. and Eng. 896 (1) 012132
[14] S Yang et al 2007 Powder Tech. 178(1) 56-72
[15] R Li et al 2010 Appl. Surf. Sci. 256(13) 4350-4356.
[16] K Kempen et al 2011 In 22nd An.Int.Solid Free. Fabric. Sym.-An Additive Manuf.Conf. 484-495
[17] D Dai and D Gu 2014 Mat. & Des. 55 482-491
[18] C Weingarten et al 2015 J.of Mat, Proc. Tech. 221 112-120
[19] I Maskery et al 2016 Mat. Charact. 111 193-204
[20] M Tang and P C Pistorius 2017 Int. J. of Fatig. 94 192-201
[21] S Lathabai 2018 Fund.of Alum. Metal. 47-92.
[22] C Weingarten et al 2015 J.of Mat, Proc. Tech. 221 112-120.
[23] S M Yusuf et al 2017 Metals 7(2) 64
[24] G Kasperovich et al 2016 Mat. & Des. 105 160-170