APPLICATION OF METALLOGRAPHY IN CHARACTERISATION OF POWDERS USED IN ADDITIVE TECHNOLOGIES

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Abstract

Powders are nowadays used as an input material not only for conventional powder metallurgy technologies, but increasingly also for additive technologies. Powder producers usually limit their description to an average chemical composition and typical particle size. However, the real particle size distribution might be much more complex than the one declared by producers and chemical composition of an individual particle also does not have to be homogeneous.

Three different powders destined for additive manufacturing were analysed, 1.2709 maraging steel, stainless steel 316L and Inconel 718. Morphology and surface appearance of particles were evaluated from scanning electron micrographs. Metallographic cross sections of powders were prepared for subsequent microstructure analysis by scanning electron microscopy (SEM) and micro-hardness measurement. Surface chemical composition of individual particles and local chemical composition of powder cross sections were checked by energy dispersive X-ray spectroscopy (EDS). Average particle size and particle size distribution were established by image analysis of SEM images of randomly chosen samples of new powders.

Keywords: Selective laser melting, maraging steel, stainless steel, Inconel

1. INTRODUCTION

Additive technologies, sometimes called also 3D printing, have been gaining increased importance in the last decade as a way of an efficient production of products with complex outer or inner geometries. There are generally more possibilities for an input material shape, whoever while plastic or composite materials could use various wires, metals are generally processed from powders [1, 2]. The nature of this processing is reflected in very unconventional microstructures of printed metals [3]. Commercially available powders of many engineering materials can be obtained for example from producers of printing devices. The powder are commonly characterises by average chemical composition and particle sizes [1]. The particle size is an important parameter not only from technological reasons but it must be also considered from health and safety point of view. The precise effect of particle size on the microstructure and mechanical properties of the final printed parts is at the moment the topic of research [4, 5] and particle shape was proved to influence powder flow during the printing process [6]. The effect of particle size on the wellbeing of operators of printing devices who are in daily contact with used powders is quite obvious, and therefore used filtering systems have to be adjusted correctly to real particle sizes. Particularly larger amounts of unexpectedly small particles of heavy metals could be dangerous, as they might avoid existing filters and could be breathed in by operators. Various works described the use of special techniques like microwave sensing method [7] and laser diffraction based methods [4,8] for particle size determination and atomic tomography was applied to provide three-dimensional models of the particles [2]. Scanning electron microscopy observations of particles were also used in studies of recycled AlSiMg powder [5] or steel powders with different chemical compositions [9, 10]. This work aims to demonstrate that methods and equipment commonly available in metallographic laboratories can be used to provide comprehensive characterisation of various powders used for additive technologies.

2. EXPERIMENTAL PROGRAM

2.1. Materials

Three powders (**Table 1**), MS 1 steel, 316 L steel and Inconel 718, were chosen for characterisation in this work with regard to various research projects and industrial cooperation solved at Regional technological Institute. All three powders were produced by gas atomization method and supplied by EOS GmbH.

Tool steel MS1 is used as the main material for design of printed advanced machining tools with inner cooling systems. Chemical composition of used powder corresponds to US classification of 18% Ni Maraging 300, European 1.2709 and German X3NiCoMoTi 18-9-5.

Inconel 718 is designed for high temperature (up to 700° C) applications in gas turbine parts, instrumentation parts, power and process industry parts etc. IN718 corresponds to UNS N07718, AMS 5662, AMS 5664, W.Nr 2.4668, DIN NiCr19Fe19NbMo3.

316 L stainless steel was chosen as a typical corrosion resistance material. It can be also defined as 18Cr-14Ni-2.5Mo. This material is used to produce various decorative objects and surgical implants, for applications in food and chemical plants or aerospace and turbine industry.

Table 1 Chemical composition of used powders in wt. %, as declared by EOS GmbH

	C	Si	Mn	Cr	Ni	Mo	Nb	Ti	Co	Al
MS1	≤ 0.03	≤ 0.1	≤ 0.1	≤ 0.5	17 - 19	4.5 - 5.2	-	0.6 - 0.8	8.5 - 9.5	0.05 - 0.15
316 L	≤ 0.03	≤ 0.75	≤ 2.0	17 - 19	13 - 15	2.25 - 3.0	-	-	-	-
Inconel 718	≤ 0.08	≤ 0.35	≤ 0.35	17 - 21	50 - 55	2.8 - 3.0	4.75 - 5.5	0.65 - 1.15	≤ 1.0	0.2 - 0.8

2.2. Characterisation of powders

The average size and size distributions of used powders were determined by image analysis of images obtained by scanning electron microscopy. Random samples of powders were placed on the stubs covered by adhesive conductive tape and placed into an EVO 25 Zeiss microscope. Images with magnification of 200x - 500x were used for image analysis; around 250 particles of each powder were evaluated using in-house developed software of image analysis called “Zrno”.

Metallographic cross sections were also prepared form each powder, by standard mounting, grinding and polishing methods. MS1 steel powder was etched in 3%Nital while Inconel and 316L powders were etched in Adler. Scanning electron microscopy was used to characterise powder cross sections and micro-hardness measurement of HV 0.01 was applied to evaluate local hardness of individual particles. Using EDS detector, chemical composition, distribution within individual particles as well as among particles of the same powder, were checked. The size of evaluated particles was also considered. Chemical composition of the smaller and coarser particles was compared for each powder.

3. RESULTS AND DISCUSSIONS

3.1. Analysis of free particles

Randomly chosen particles were placed on the stub and analysed in SEM. The surface of individual particles was observed at high magnifications (**Figure 1 - Figure 3**). In all powders, variations of powder particles shapes were observed, from spherical individual particles, to elongated particles, bonded particles, fractured parts and smaller satellites attached to the main particles. Attachment of small particles to larger ones is a typical feature of powders produced by gas atomisation and was described by other works as well [2]. However, these morphologies were present in individual powders with different frequencies and some differences were observed between the powders. MS1 particles possessed mainly relatively regular, spherical shapes with the structure of dendritic arms or cells clearly visible on the whole surfaces. Fine spherical satellites were often attached to larger particles of regular shapes.

On the other hand, the particles of Inconel 718 had much more complicated surface geometries. There were many “ragged” particles without any defined shape and spherical particles with visible parts of some accretionary surface envelopes were found in this powder. These envelopes had always different, smoother, surfaces without traces of any dendritic or cellular structure. 316 L steel powder consisted of the largest particles with mainly regular spherical shape and occasional parts of broken “ragged” particles. All particles showed dendritic or cellular structure on the surface.

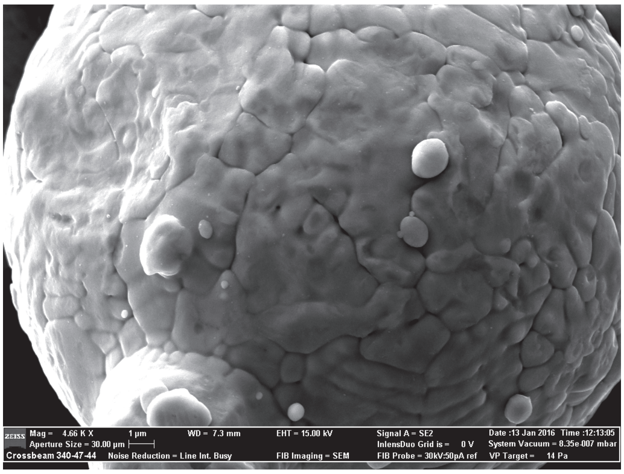

Figure 1 MS 1 powder morphology

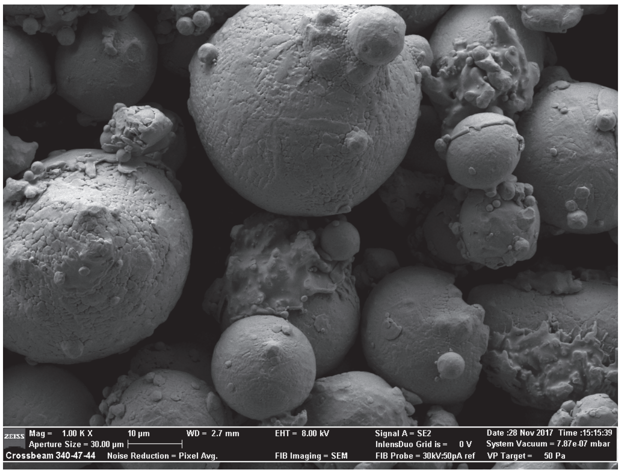

Figure 2 Inconel 718 powder morphology

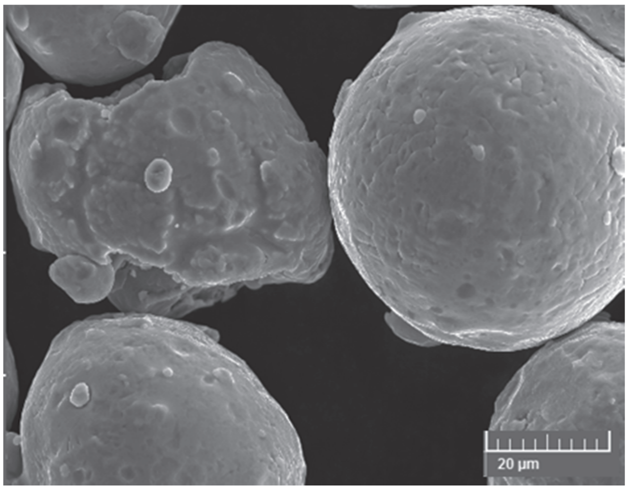

Figure 3 316L powder morphology

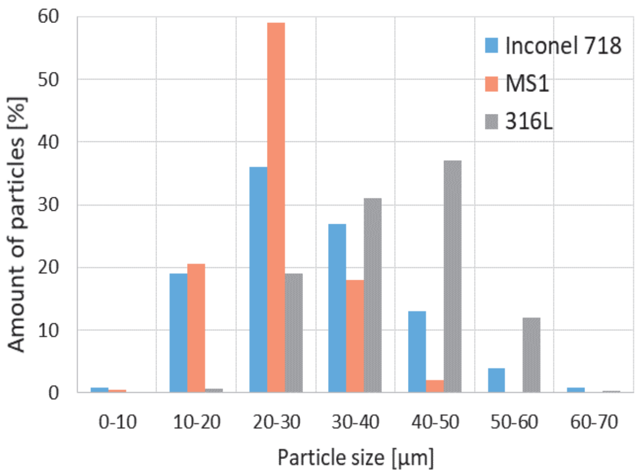

Figure 4 Particle size distributions

Average particle size and particle size distributions were also calculated for each material. **Figure 4** shows how many particles (in percent) fall within 10 µm size intervals (i.e. up to 10 µm, between 10 µm and 20 µm etc.). It is obvious that most of the particles of MS 1 and Inconel 718 had the sizes around 20 - 30 µm. Powder of Inconel 718 however provided flatter distribution curve, meaning that there were more particles of larger sizes than in the case of MS 1 steel. The average particle size of Inconel 718 is therefore higher, reaching 30 µm, while MS 1 steel possessed the finest average particle size of only 24.8 µm. The particles of 316 L powder were the coarsest ones, with the average size of 39.4 μm. The distribution curve of 316 L steel was significantly shifted to the right with most of the particles having the sizes around 40 μm.

For all three materials, surface chemical composition of large and small powder particles was measured for a comparison. There wasn't any detectable difference for Inconel and 316 L powder, however in the case of MS 1 steel, smaller particles, with sizes around 10 μm, tended to have slightly lower cobalt and molybdenum contents and higher nickel content than the largest particles. It is also interesting to note, that form 25 analysed particles of MS1, there were three particles with increased chromium content, which in one case reached 14%. Inconel 718 and 316 L steel didn't show any difference in chemical compositions of large and small particles of the same powder. However, smooth envelopes were observed on the surface of some particles of Inconel 718, regardless the size of the particles. The envelopes generally possessed higher contents of the main alloying elements such as nickel, chromium, iron and manganese, while lacking niobium and molybdenum. In several cases, oxygen was detected in increased amounts in these smooth layers. 316 L steel particles did not display any anomalies in chemical composition and there were not apparent differences between fine and large particles or various surface areas.

3.2. Analysis of metallographic cross sections

Chemical composition was also locally measured at metallographic cross sections of powders in an attempt to distinguish chemical compositions of the cells or dendrites and cell boundaries or inter-dendritic spaces. Powders of all the three materials contained the mixture of dendritic and cellular microstructures (**Figure 5 - Figure 9**). Various defects were also observed, for MS1 there were spherical inclusions with increased iron and titanium contents (**Figure 6**). Boundaries of the cells had slightly different chemical composition than the matrix particularly molybdenum content was higher in the boundaries. It was also detected that finer cells had slightly higher molybdenum contents than the larger cells (**Figure 6**). This could have been caused by stronger effect of high molybdenum boundaries on values obtained in smaller cells, rather than by really higher molybdenum contents inside smaller cells. Micro-hardness was measured at individual particles; the average value calculated from ten particles was 336 HV0.01. Measured values were in the range of 286-357 HV 0.01.

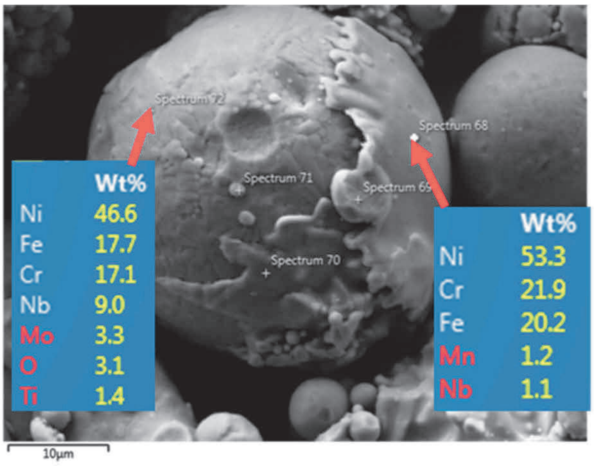

Figure 5 Chemical composition of surface envelope and the particle, Inconel 718

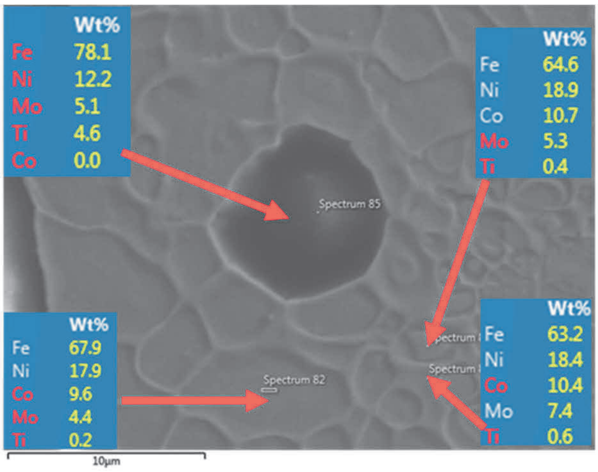

Figure 6 Cross section of MS 1 particle with inclusion, local chemical compositions

Inconel 718 also had a distinctive dendritic and cell microstructure (**Figure 7**), but it was difficult to quantify conclusively differences in chemical compositions of the matrix and the boundaries, as there was quite a big scatter of contents of individual elements and a rather fine microstructure. Microhardness of individual particles was in the region of 328 - 354 HV 0.01, the average being 340 HV 0.01.

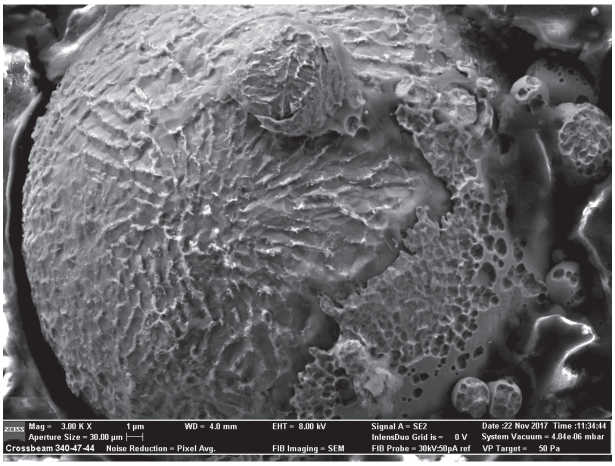

Figure 7 Cross section of Inconel 718 particle

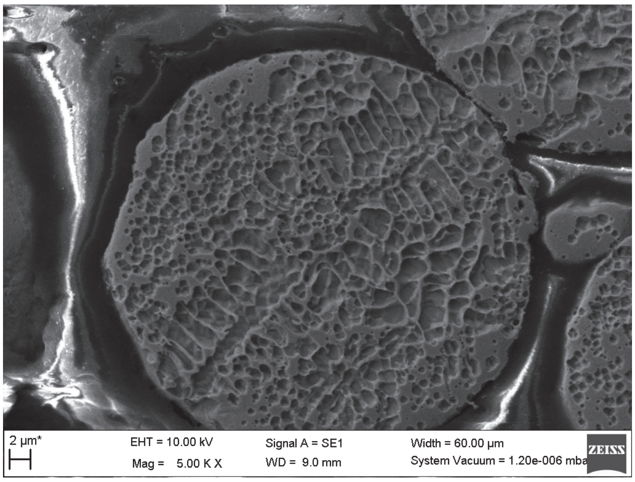

Figure 8 Cross section of 316 L particle

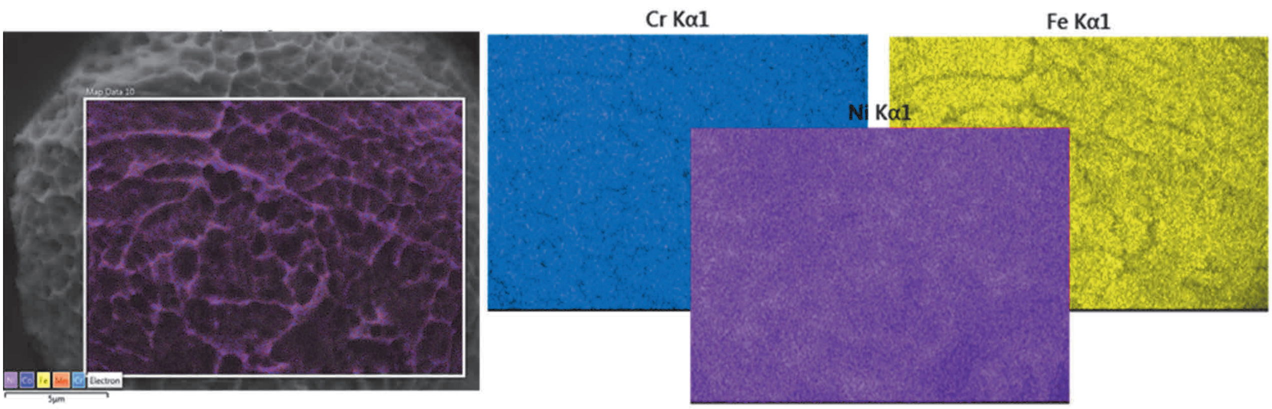

Figure 9 Elemental distribution in 316 L particle

316 L steel particles contained a dendritic microstructure (**Figure 8**, **Figure 9**) with very similar chemical compositions of the matrix and inter-dendritic boundaries. There were only slightly higher nickel contents at the boundaries than inside the dendrites. This tendency was clear not only from point measurements, but also from element mapping (**Figure 9**). Microhardness of the particles was the lowest one of the three materials, lying in the interval of 169 and 288 HV 0.01 and reaching the average hardness of only 226 HV 0.01. These results are in agreement with micro indentation measurements carried out at 316 L steel powder by Azevedo et al [9].

4. CONCLUSION

Powders of three different metals prepared by gas atomization and intended for further processing by additive manufacturing were characterized by the means of metallography, micro-hardness measurement, scanning electron microscopy and local measurement of chemical compositions. Different distribution of particle sizes were obtained with MS1 steel possessing the finest powders of 24.8 µm, Inconel 718 having generally more particles of slightly larger size and average size of 30 µm and 316 L steel showing the biggest portion of coarse particles and average size of 39.4 µm.

Mixtures of dendritic and cellular microstructures were observed in all three powders. Chemical composition of the cells or dendrites boundaries differed from the composition of the matrix for MS 1 steel and 316 L steel. Increased molybdenum content was detected in the boundaries of MS1 particles and increased niobium content in the case of 316 L steel. No conclusive results were obtained in this regard in Inconel 718. On the other hand, Inconel 718 displayed parts of smooth envelopes attached to the surfaces of some particles. There was higher content of Ni, Cr and Fe in these envelopes than in the rest of the particles and the amounts of minor alloying elements were lower. Average micro-hardness of MS1 steel powder was 336 HV 0.01, Inconel 718 reached very similar value of 340 HV 0.01, while 316 L steel achieved the lowest hardness of 226 HV 0.01.

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