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Powder for Metal Additive Manufacturing: Production, Reuse and Recycling

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Abstract
Metal powder is the feedstock for most of the metal additive manufacturing (AM) technologies, including powder bed fusion – laser beam (PBF-LB) and electron beam (PBF-EB), binder jetting (BJT) and powder blown directed energy deposition (DED). However, even if nearly the same alloys systems are used, requirements to the powder feedstock are rather different. Processing conditions during powder-based metal AM differ significantly, depending on technology, hardware solution and process parameters employed. This results in changes in powder properties during manufacturing cycle and especially during its reuse, also having significant impact on the final component properties. This work summarizes recent experimental observations and thermodynamic simulations of the changes in powder properties during the whole life-cycle of metal powder: from its manufacturing through powder handling and AM processing by variety of powder-based metal AM technologies. Generic model of the powder degradation in dependance on alloy composition and AM technology, is elaborated.

Keywords: metal additive manufacturing, powder for AM, powder manufacturing, powder degradation, powder reuse.

Introduction

Additive manufacturing (AM) is a revolutionary technology that is believed to alter the manufacturing and logistic landscape, having significant impact potential in reaching the common climate goals and at the same time increasing manufacturing competitiveness. There is huge number of commercialized AM technologies on the market nowadays that, according to ISO/ASTM 52900/2021 standard[1)], can be categorized into seven AM technology groups, namely binder jetting (BJT), directed energy deposition (DED), material extrusion (MEX), material jetting (MJT), powder bed fusion (PBF), sheet lamination (SHL) and vat photopolymerization (VPP). Over the last decade, significant efforts were focused on the development of metal AM technologies, where metal powder is the basic feedstock for metal AM for all mentioned above categories of AM technologies, except SHL. Some metal AM technologies are just on rise, as e.g. MJT, VPP and MEX, that are using fine, often nano-powder, followed by sintering, providing new possibilities for material prototyping or manufacturing of high-end small-size metal components. Powder-based metal AM technologies as PBF, MJT and DED provide the core of the metal AM and already secured their position in industrial manufacturing of high-performance metal AM components for medical, aerospace, energy, structural and consumer sector, etc.

Even though metal powder provides the basic material feedstock for most of the metal AM technologies, there is a lack of understanding of the effect of powder properties on suitability of the powder for a specific AM process, meaning relationship between process parameters and powder properties. This negatively impacts process robustness, reproducibility and final material properties. At the same time, powder can be manufactured using variety of powder manufacturing methods, ranging from well-controlled powder atomization methods allowing to produce powder of high sphericity and purity, as e.g. Electrode Induction-melt Gas Atomization (EIGA), Plasma Rotating Electrode Process (PREP) and Plasma Atomization (PA) processes. Powder produced by these methods is a bit coarser (EIGA and PREP), is produced by ceramic-free melting and is often used for manufacturing of high-purity or reactive alloys, as for example Ti- and Ti-alloys, TiAl, Al-alloys, Ni-base superalloys, precious metal powders, etc[2)]. Powder produced by these technologies is the most used material feedstock for high-end applications requiring high purity as e.g. medical and aerospace, and hence provide the base material feedstock for DED, powder bed fusion – electron beam (PBF-EB) and to some extent in powder bed fusion – laser beam (PBF-LB). The disadvantage of these methods is low productivity and hence the high cost of the metal powders. When it comes to the processes requiring powder with finer particle size, as e.g. powder bed fusion – laser beam (PBF-LB) and BJT, that are also more sensitive to the cost when it comes to e.g. structural applications, tooling, automotive applications, etc., more large-scale inert gas atomization methods[2)], as e.g. inert gas atomization (GA) and Vacuum Induction Gas Atomization (VIGA) are used, the later one providing better quality metal powder with reasonable costs.

However, even though similar/the same powder feedstock is used by number of AM technologies, powder consolidation during AM processing differs significantly between different AM technologies. Interaction between top surface layer of powder with intense beam of photons produced by powerful lasers in PBF-LB and DED is very different in comparison to very similar but at the same time very different PBF-EB technology, where high-energy electrons penetrate much deeper into the powder particles and result in totally different melting mechanism[3)]. At the same time, when it comes to the binder jetting (and other sinter-based technologies), powder consolidation is taking place through inter-particle sintering. Sintering is characterized by much lower process kinetics and requires efficient removal of binder residues and surface oxide layers to assure efficient densification and purity of the final component[4,5)].

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In powder-bed additive manufacturing processes, up to 80% of the powder volume in the build chamber is not used for component manufacturing and hence should be reused in order to assure necessary process efficiency and economic feasibility of AM process. At the same time, there is a very limited understanding of the powder degradation during AM process, especially relationship between powder chemistry, AM process parameters and final impact of powder degradation on the AM processability of the powder, process stability and robustness, final microstructure and material properties of the material. Therefore, most of the powder handling and powder reuse approaches are typically based on evaluation of powder physical properties, as e.g. flowability, and/or powder chemistry in terms of changes in bulk oxygen, or even carbon and nitrogen, depending on material and application.

It is important to note that metal powder used for AM is very chemically active material, characterized by a surface area that is about 10 000 times larger than the surface area of the bulk material of the same mass in case of powder typically used for PBF-LB. This leads to the inherently higher oxygen content in the metal powder due to the naturally present metal oxide, ranging between 1 and 10 nm, depending on the material and manufacturing method[6-10]. However, surface oxide, covering metal particles, is unstable and can change significantly during handling as well as during AM processing. Changes in surface oxide chemistry are determined by the thermodynamic stability of the surface oxide, present on the metal powder in as-atomized state from one hand, and exposure to the elevated temperature as well as oxygen potential during exposure and time of the exposure. Hence, temperature and time of the exposure in combination with the oxidation potential of the processing atmosphere determines chemical composition, thickness and distribution of the oxide phases on the powder surface.

Paper provides overview of the numerous studies performed at the Centre for Additive Manufacturing – Metal (CAM^2)[11], focusing on impact of powder manufacturing on powder characteristics and powder behavior during AM processing, interaction between process parameters and metal powder, and especially powder degradation in dependance on alloy composition, additive manufacturing process parameters as well as its impact on final microstructure and final properties of the components.

Experimental Procedure

A variety of gas atomized powders were studied, as summarized in Table 1. For PBF-LB, powders with a size between 20 and 53 μm were used, except AlSi10Mg where powder with size of 45 to 105 μm was used. In case of In718 powder studied by PBF-EB, standard size fraction between 45 to 105 μm was used. Standard process parameters, provided by technology providers for each of the studied materials, were used.

Table 1. Powder studied as well as AM processes applied

Powder	Powder atomization	AM process	Hardware
316L	GA	PBF-LB	EOS M290
316L	VIGA	PBF-LB	EOS M290
AlSi10Mg	GA	PBF-LB	XLINE 2000R
In718	VIGA	PBF-LB	EOS M290
In718	PA	PBF-EB	Arcam A2X

Powder samples were prepared by mounting the experimental powder on a carbon tape prior to analysis by X-ray photoelectron spectroscopy (XPS). For the HR SEM + EDX analysis of the powder, powder samples were prepared by lightly pressing the powder onto soft aluminum plates. The surface chemical analysis of the powder was performed by means of X-ray photoelectron spectroscopy (XPS) using a PHI 5500 instrument (Perkin Elmer, Waltham, Massachusetts, USA). The analyzed area during XPS analysis was about 0.3 mm in diameter. Hence, about 10 to 100 powder particles were analyzed at the same time giving statistically reliable average results that represent the general powder surface composition. Photoelectrons were generated by monochromatic Al Kα source (1486.6 eV). Selected region spectra were recorded covering the peaks of interest. The acquisition conditions for such high-resolution spectra were 23.5 eV pass energy with the step of 0.1 eV and nominal take-off angle of 45°. The recorded photoelectron peaks were curve fitted utilizing the PHI Multipak software (Perkin Elmer, Waltham, Massachusetts, USA). Quantification of the results was performed by calibration measurements on pure elemental standards. Determination of the surface oxide layer thickness and compositional profiles was done by alternating ion etching and XPS analysis. Evaluation of the thickness of the surface oxide layer was performed using theoretical model, established for XPS analysis by host group[12-14], that takes into consideration rough surface of the powder sample as wall as geometry of the XPS setup. Model is integrated in the software "Powder XPS calculator v.1.2.4", developed by authors for powder analysis. Powder morphology as well as surface oxide distribution and morphology was studied by high-resolution scanning electron microscope (HR SEM) LEO Gemini 1550, equipped with INCA Energy/X-Max analyzer.

Results and Discussion

Stainless steel 316L is the most used powder in PBF-LB process due to its wide range of utilization, high processability and robustness by PBF-LB as well as excellent material properties[15-17]. In addition, powder can be produced with the very good quality by variety of methods[6], especially considering low-cost high-productivity atomization technologies ranging from water atomization, gas atomization and VIGA, see Fig.1 (a-d). The as-atomized powder, produced by either GA or VIGA, is covered by a homogeneous iron oxide layer, formed by Fe_2O_3, with a thickness of about 4 nm. Nano-sized oxide particulates, see Fig.1 (b and d) are rich in Cr, Mn and Si and their fraction and size is higher in GA compared to VIGA[6]. Even though 316L is characterized by high robustness in PBF-LB, powder degradation is inevitable during PBF-LB processing due to the spatter formation and accumulation, as is revealed by the presence of highly oxidized particles in the reused powder, see Fig.1 (e-h), especially when processed in the atmospheres with the higher oxygen potential[17], as e.g. the one produced using nitrogen generator, see Fig.1 (g and h) characterized by oxygen content of 10 000 ppm compared to conventionally used 1000 ppm in Ar, see Fig. 1 (e and f).

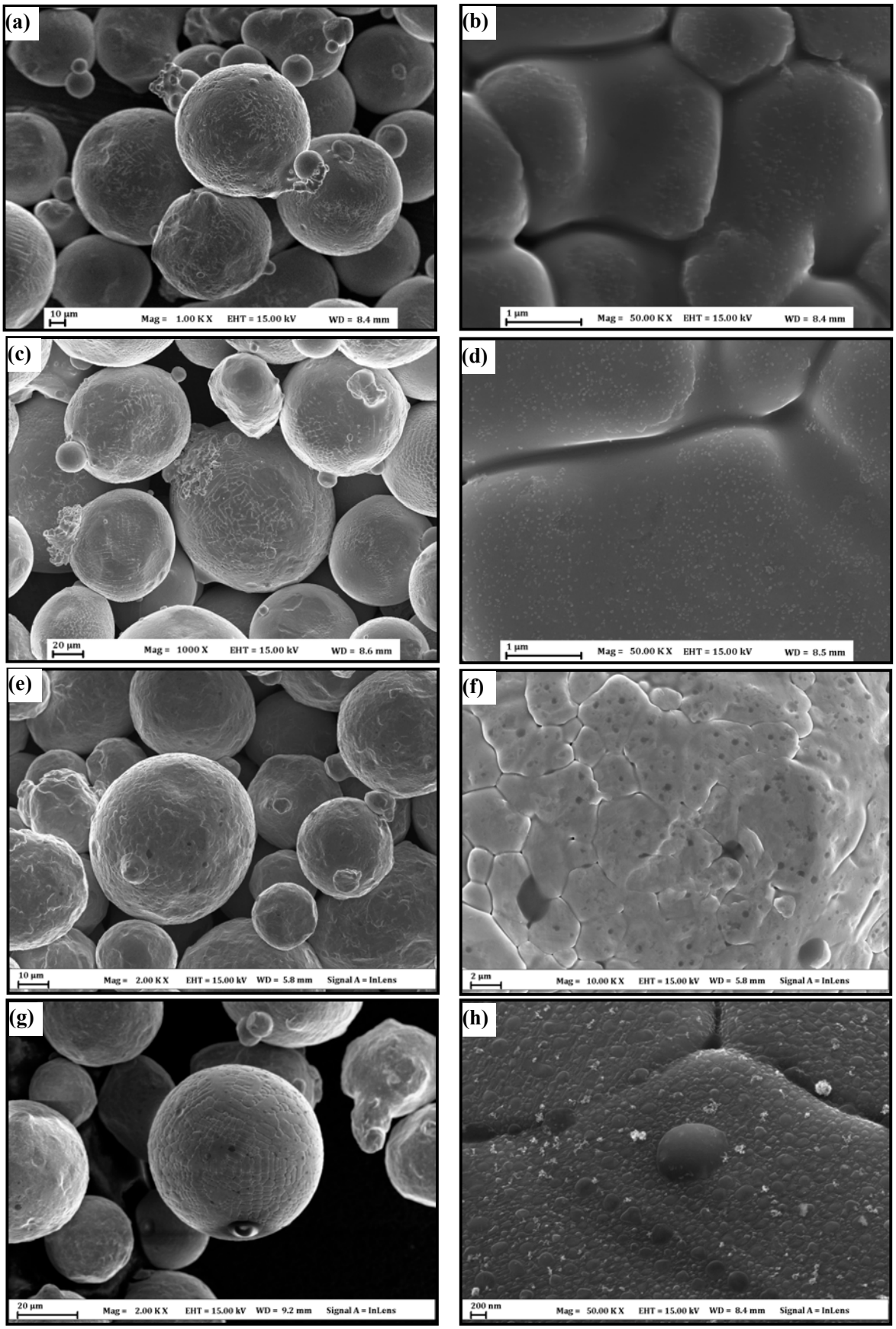

Fig.1. SEM micrographs of 316L powder in as-atomized state, produced by conventional gas atomization (a,b) and VIGA (c,d). Powder degradation after PBF-LB processing in Ar (e,f) and nitrogen with high oxygen potential (g,h).

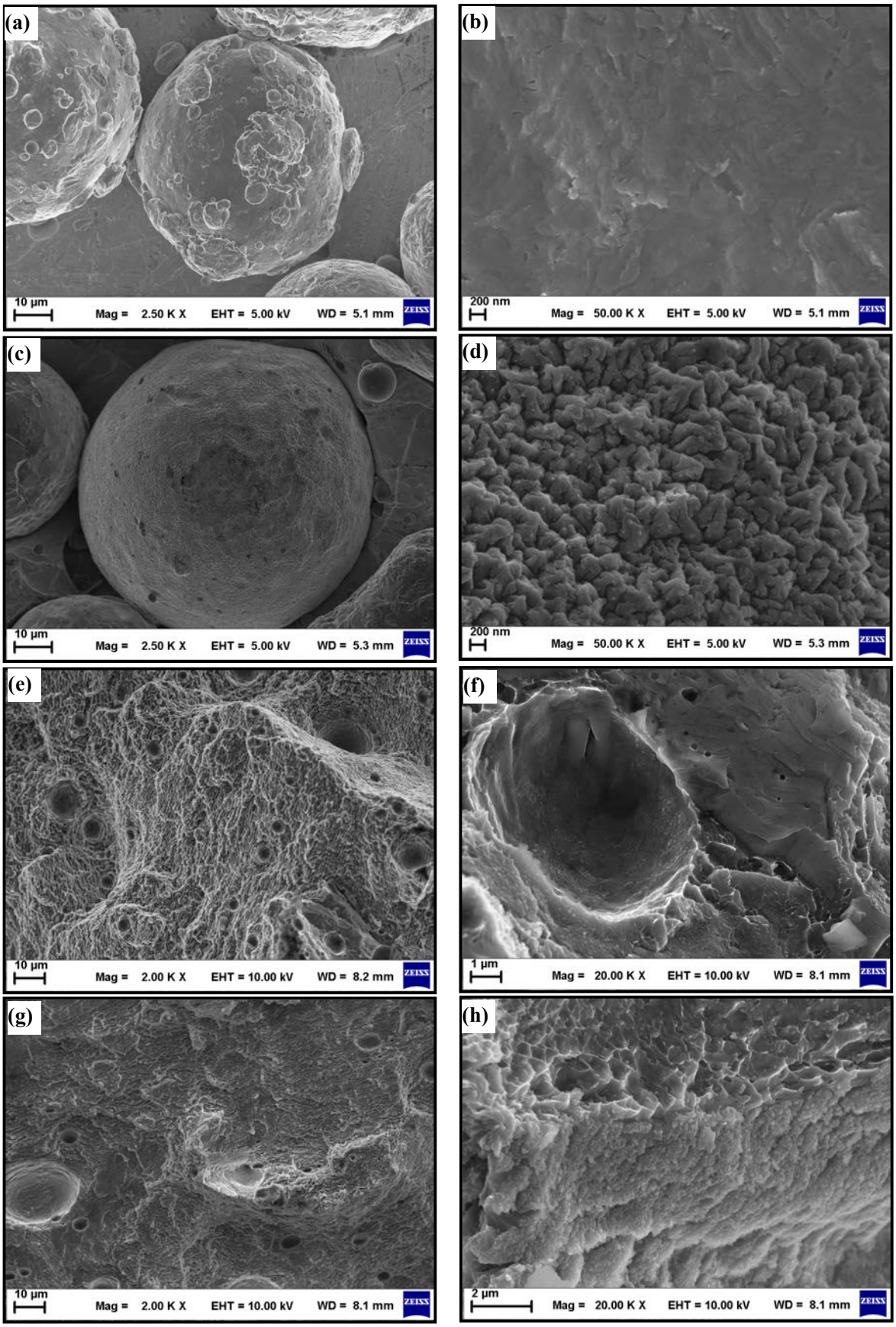

Fig.2. SEM micrographs of Al10SiMg powder in as-atomized state (a,b), powder after PBF-LB processing for 30 month (c,d), and fracture surface of PBF-LB processed components from virgin (e,f) and reused for 30 month powder (g,h).

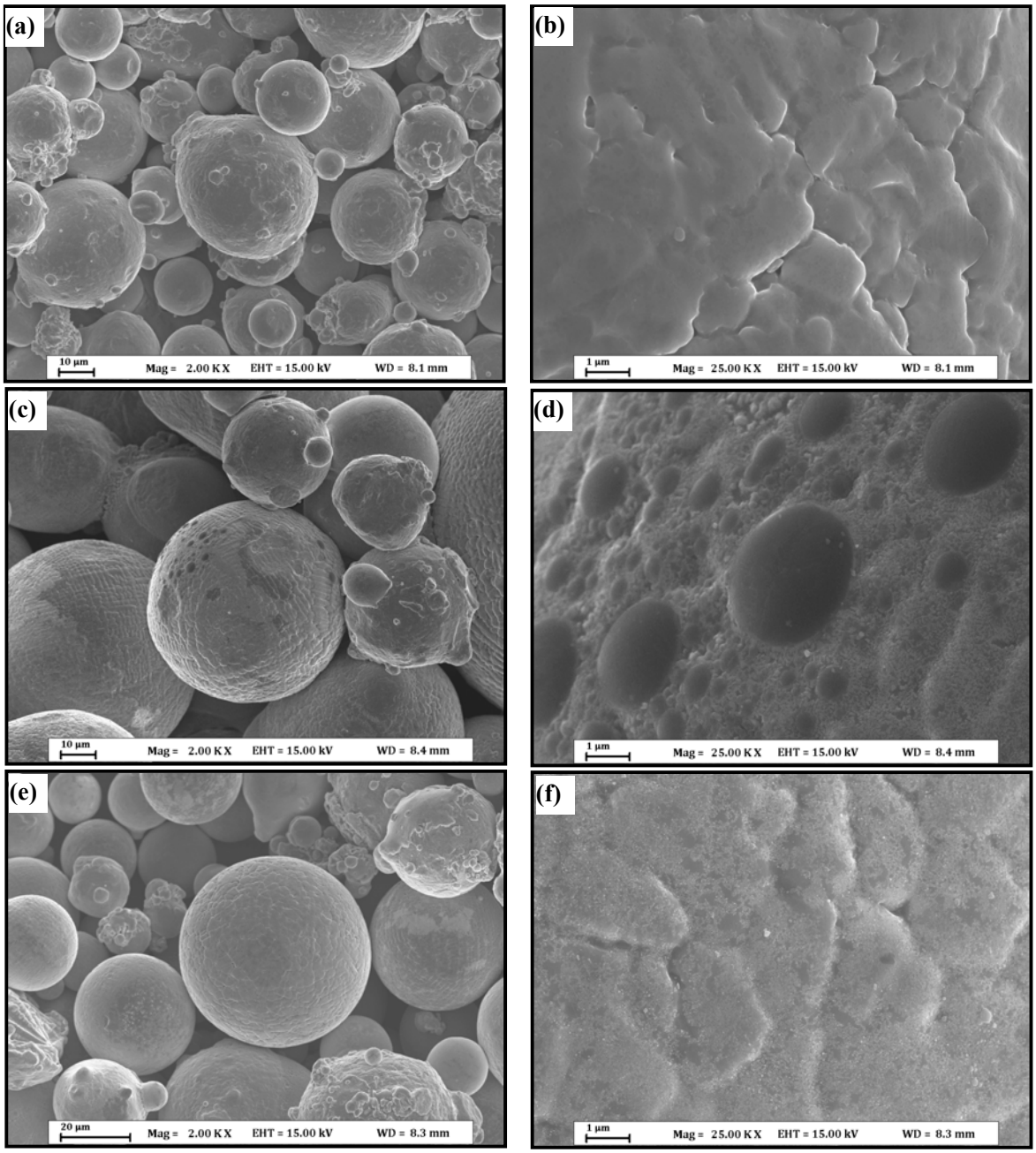

Fig.3. SEM micrographs of In718 powder in as-atomized state, produced by VIGA (a,b), as well as example of particles after one reuse cycle during PBF-LB processing in Ar-atmosphere with 1000 ppm O_2(c,d) and ~20 ppm O_2 (e,f).

Al10SiMg powder is characterized by much higher stability of oxide layer, formed by Al_2O_3 with the thickness of about 4 nm in case of the virgin as-atomized powder[18]. Powder degradation in case of PBF-LB processing is strongly connected to the spatter accumulation, with the spatter particles having characteristic nodular surface oxide scale, see Fig.2 (c,d), thickness of which is in the range of 50-125 nm[18], compared to the relatively smooth surface in case of the virgin powder, see Fig. 2 (a,b). The volume fraction of spatter particles increases up to around 3% after powder reuse for 30 months[18], that has negative impact on final porosity and mechanical properties of the produced components[19]. The porosity in the case of components produced using reused powder is about 4 times higher in comparison to the samples produced from the virgin powder at the same hydrogen content in both materials[19]. Analysis of the fracture surface, see Fig.2 (e-h), clearly show presence of higher porosity in case of the samples made of reused powder with the presence of oxide scale, see Fig.2 (g,h), with the same morphology and nature as was observed on the surface of the spatter particles, see Fig.2 (d). The mechanical performance of the specimens produced from reused powder was also decreased, with tensile strength, yield strength, elongation at break, and microhardness being reduced to up to 15 %[19].
Hence, it is clear that spatter formation and its further oxidation is the main degradation mechanism in the case of PBF-LB[18-22]. As spatter formation is not possible to avoid due to the nature of laser-powder interaction in the presence of the processing gas, the most feasible mechanism to minimize impact of the spatter particles, in addition to the possibility to minimize spatter formation by

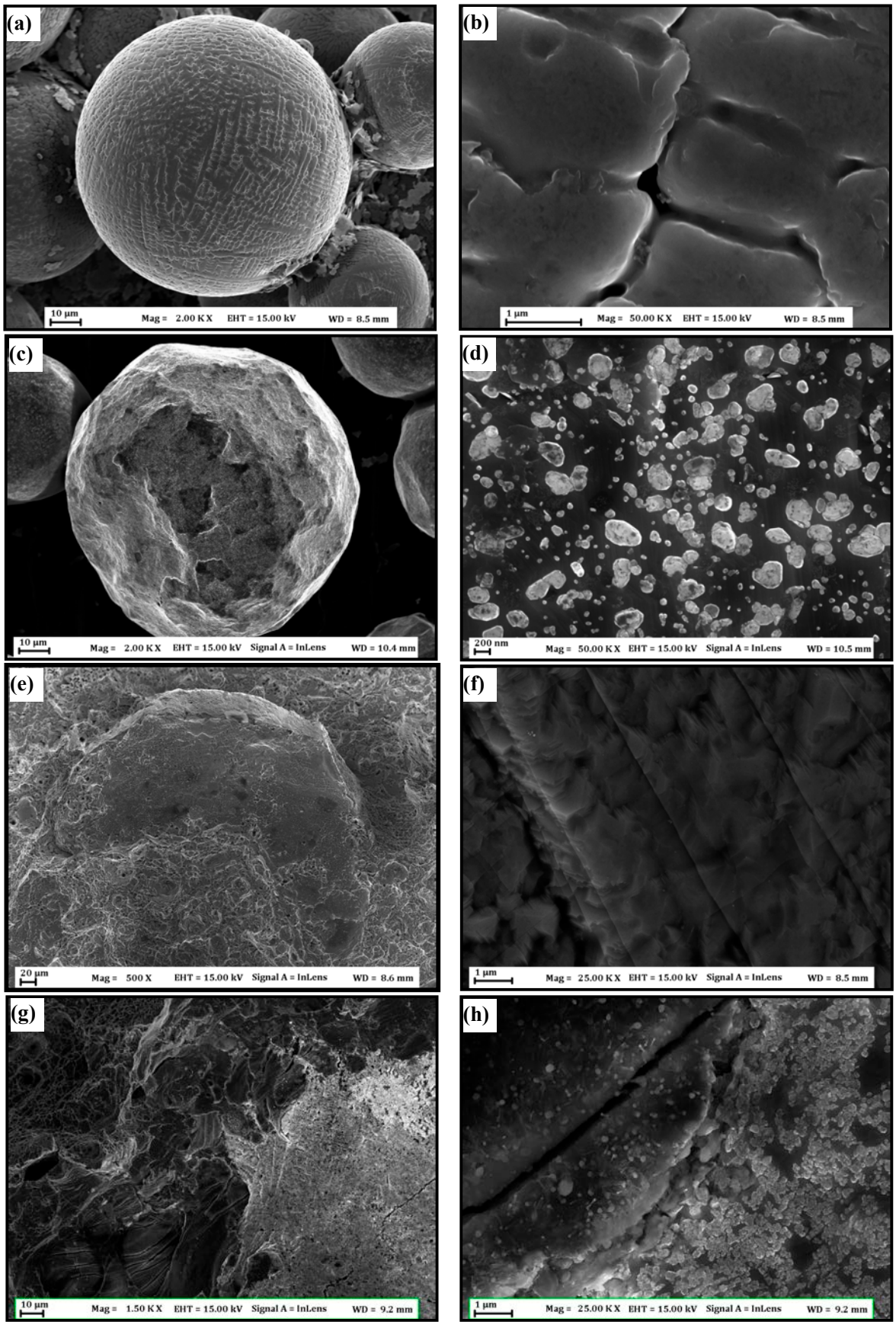

Fig.4. SEM micrographs of In718 powder in as-atomized state, produced by plasma atomization (a,b), powder degradation after PBF-EB processing for 13 cycles (c,d), and fracture surface of PBF-EB processed components from virgin (e,f) and 40-times reused powder (g,h).

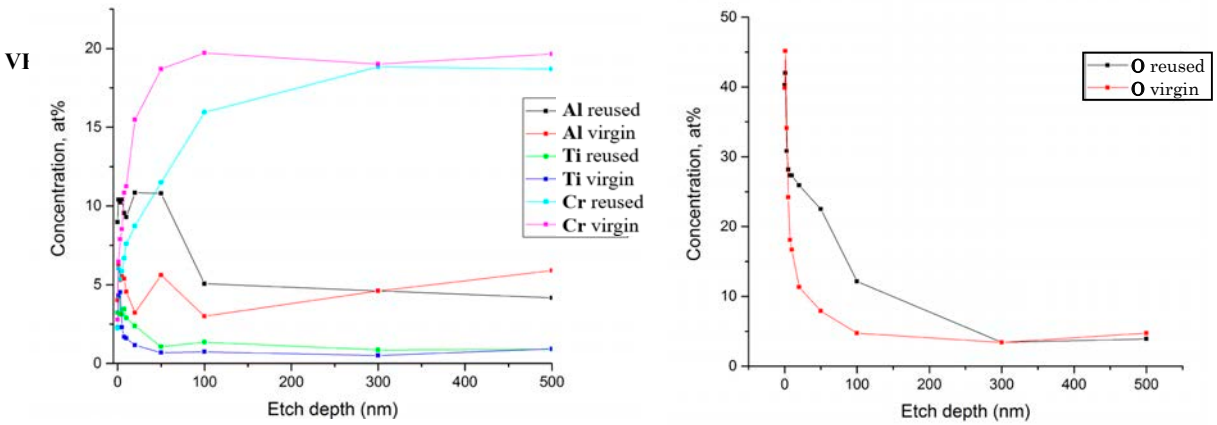

Fig. 5. XPS analysis of In718 virgin and reused (14x) powder during PBF-EB processing, indicating enrichment of Al and Ti in reused powder (left) as well as increase in oxide layer thickness (right).

improving powder properties, optimization of process parameters and surface-to-volume ratio of the components[20-24], is to minimize oxidation potential during PBF-LB processing[25]. Even though very good quality powder in case of In718 can be obtained by VIGA, see Fig. 3 (a,b), processing under conventionally used Ar-atmosphere with 1000 ppm of oxygen results in rather significant oxidation of spatter particles, see Fig. 3 (c,d), that are depositing on the powder bed as well as printed components and can lead to the defect formation, especially in case of higher energy input, higher layer thicknesses or high surface to volume ratio, as e.g. printing cellular structures[21-24]. Decreasing oxygen content in the processing gas allows to avoid/minimize spatter oxidation, see Fig. 3 (e,f) and hence minimize/avoid impact of spatter on process stability and material properties as well as to significantly improve powder reusability[25].

When it comes to the PBF-EB, process is typically performed at higher layer thicknesses and hence allows to use coarser metal powders. Considering aerospace applications, most typical powder used for PBF-EB is produced by plasma atomization and is characterized by high sphericity and excellent purity, see Fig. 4 (a,b). Distinguishing feature of PBF-EB processing is that process is executed in high vacuum to avoid scattering of electron beam and at elevated temperature to ensure powder pre-sintering and necessary process stability. Even though high vacuum, on the level of 10^{-6} bar, is positive for minimizing powder surface oxidation, high processing temperature, that is at around 1000 °C in case of processing of In718, can lead to significant powder oxidation, see Fig. 4 (c,d), especially considering long building times, that can exceed 100 h for one build cycle. Selective oxidation of Al, leading to formation and accumulation of nanosized Al-rich oxide particulates on the powder surface can be seen[26], see Fig.4 (c,d). This is also confirmed by XPS analysis, see Fig.5, where significant enrichment in Al can be clearly seen. A slight increase in Ti can be also registered, whereas Cr content is lower that alloy content on the top powder surface due to the significant enrichment in more stable Al-based oxide. The presence of heterogeneous oxide layer can be seen based on the oxygen profile as well, see Fig. 5 (right), where presence of thin Ni-based oxide layer is similar for virgin and reused powder. However, there is a steep deviation in oxygen profile up to 100 nm etch depth, indicating presence of particulate oxide features with the size up to 100 nm, that is in agreement with the SEM observations in Fig. 4 (c,d).

Enclosure of observed oxide inclusions in PBF-EB processed components lead to increase in the bulk oxygen content in the produced components[26], which increases with progressive re-use. Clear impact of oxide inclusions on the defect formation can be seen as well when comparing rather defect-free components produced from virgin powder with clean interfaces[27], Fig.4 (e,f), with microstructure and fracture surface of the components, produced from reused powder, where clear presence of oxide agglomeration in defect sites can be seen, Fig. 4 (g,h). Hence, it can be concluded that the main source of oxide defect formation in the PBF-EB fabricated samples is the Al-rich oxide originating from the surface of the re-used feedstock powder, which tends to cluster during consolidation. When it comes to the spatter formation and its impact in PBF-EB[28], spatter oxidation is not as big an issue as in case of PBF-LB due to significantly lower oxygen potential. However, sublimation of alloying elements and its impact on powder reuse is of a bigger challenge [28].

Conclusions

Powder surface chemistry is determined by powder atomization process as well as alloy composition. Even though high-quality powder is obtained using well-established powder atomization processes, powder is very sensitive to powder handling and reuse. Reusability of the powder is strongly determined by the alloy composition, surface oxide present on the powder as result of the powder atomization and handling, as well as powder exposure during AM process in terms of time, temperature and oxygen content. In the case of powder bed fusion – laser beam, powder degradation is connected to the formation and strong oxidation of the spatter particles and their further accumulation in the powder bed and hence reused powder. Heavily oxidized powder particles lead to defects in as-printed material as e.g. inclusions, lack of fusion defects and porosity, resulting in inferior properties of the produced parts. Degradation in case of powder bed fusion – electron beam is connected to the long-term exposure of the powder in the powder bed to the elevated processing temperature, that even when processed in relatively high vacuum, can lead to extensive powder

degradation due to the relatively high oxygen potential in the processing chamber.
Hence, it can be concluded that powder surface chemistry undergoes significant changes during AM processing and has an important impact on powder processability by additive manufacturing. Hence, it must be well understood to assure necessary process robustness and final microstructure and properties of AM components.

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