Anisotropy in local microstructure – Does it affect the tensile properties of the SLM samples?

T. Maity\textsuperscript{a}, N. Chawake\textsuperscript{b}, J.T. Kim\textsuperscript{b}, J. Eckert\textsuperscript{a,b}, K.G. Prashanth\textsuperscript{b,c,*}

\textsuperscript{a}Department of Materials Physics, Montanuniversitat Leoben, Jahnstrasse 12, A-8700 Leoben, Austria
\textsuperscript{b}Department of Manufacturing and Civil Engineering, Norwegian University of Science and Technology, Teknologivegen 22, 2815 Gjovik, Norway
\textsuperscript{c}Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Jahnstrasse 12, A-8700 Leoben, Austria

1. Introduction

Additive manufacturing (AM) techniques such as laser based powder bed fusion process (selective laser melting – SLM) has attracted increasing attention because of their ability to fabricate near-net-shaped engineering and structural components of theoretically any shape with added functionality [1–3]. SLM process offers a very high cooling rates in the order of $10^5$–$10^6$ K/s that helps in developing very fine microstructures, whereas, the layer by layer operation allows the manufacturing intricate and complex structures with high accuracy [4–9]. The samples produced by SLM show improved properties if not matches the properties of the counterpart produced by conventional casting/powder metallurgy techniques [10–13]. Several metallic systems including Al-based alloys [14–18], Ti-based alloys [19–22], Fe-based alloys [23–25], Ni-based alloys [26–28], etc. have been studied in detail. Among these alloys, Al-Si family and its composites have gained considerable interest because of their excellent properties like weldability and fluidity [29–33].

Since SLM process offers a very high cooling rates [6], a very fine cellular microstructure is observed in the SLM processed Al-12Si samples with Al along the core of the cells and Si along the cell boundaries [34,35]. In addition, the microstructure is observed to be non-homogeneous, with differences in features observed along the hatch overlaps and along the core of the hatches [10,14]. Having such inhomogeneous microstructure may lead to difference in their mechanical properties. Hence, there needs a clear understanding of the local mechanical properties at nano-scale, which may be linked to the microstructure and changes in the local chemical composition. A sound knowledge on the homogeneity of the mechanical properties of the materials both at different length scales are imminent to fully realize the potential of the SLM process. Everitt et al. has reported that the local mechanical properties are uniform for SLM manufactured AlSi10Mg using the nano-indentation technique [36]. However, that is the only work reported and on a single material (AlSi 10 Mg), which deals with the local mechanical properties of SLM processed materials.

Hence, the present work is another attempt to study the local mechanical properties of SLM processed Al-based alloy. Nano-indentation investigations have been performed on the SLM processed Al-12Si alloy to explore the local mechanical properties, since nano-indentation is one of the depth sensing techniques that is capable of studying mechanical properties of materials at small-scale volume, by determining nano-hardness ($H$) and Young’s modulus ($E$) [37,38]. However, application of nano-indentation technique in SLM processed materials are not common until now and the potential of nano-indentation has been effectively utilized to demonstrate the local mechanical properties of SLM processed materials.
Al-12Si alloy. The nano-indentation results were compared with the micro-indentation and tensile test data.

2. Experimental

Al-12Si SLM samples were fabricated using Concept Laser M2 device (equipped with 400 W fiber laser) with the standard parameter set from the device manufacturer (laser power: 380 W, scanning speed: 1525 mm/s, layer thickness: 30 μm, laser spot size: ~80 μm and hatch distance: 100 μm). The samples were prepared over an Al-12Si cast substrate plate with support structures in between them. The Al-12Si specimens were processed under inert Ar atmosphere to avoid any oxidation during the fabrication process. After the processing, the specimens were mechanically polished using conventional grinding and polishing steps. The final polishing to mirror finish was carried out using diamond polishing.

Microstructural investigations were performed on etched condition (etchant – Keller’s reagent: 5 ml nitric acid, 3 ml hydrochloric acid, 2 ml hydrofluoric acid, and 190 ml distilled water) with Olympus (BX51) light microscope and a Zeiss 1525 scanning electron microscope (SEM) equipped with energy dispersive X-ray (EDX) and electron back scattered diffraction (EBSD) setups. Hardness testing was carried out using a Buehler (Micromet 5104) nano-indenter with 10 gf load, and a dwell time of 10 s was used.

Nano-indentation was carried out using a Hysitron Triboindenter device with a 3 sided pyramid Berkovich tip with a 260 nm radius (curvature) according to the ASTM E 2546 at load-controlled mode (CLR). The load was increased at 0.375 mN/s up to a maximum load (Pmax) of 12 mN followed by a 10 s holding time. The unloading was carried out at the same rate (0.375 mN/s). Indents were typically made to a maximum depth (hmax) = 500 ± 15 nm at Pmax = 12 mN. The nano-indentation data were analyzed using standard Oliver and Pharr techniques to determine H and E for each indentation [38-40]. Fused silica was used for calibrating the tip area function. The tests were carried out until the thermal drift was reduced to ~0.05 nm/s. The drift correction and indentation strain rate has been estimated according to Mayo and Nix [38]. The pre- and post-indentation drift along with indenter-tip shape factor was taken into account, while analyzing the data after taking 300 indentations in an array.

The nanohardness (H) of specimens were calculated from the unloading curves using the following Eq. (1) derived from Hertzian theory of contact mechanics [35-38]:

\[ H = \frac{P_{\text{max}}}{A_e} \]

where \( P_{\text{max}} \) is maximum applied load and \( A_e \) is the contact area/indentation area function. The indentation contact depth \( h_e \) was determined by fitting a polynomial expression to the upper 70% of the unloading curves. \( A_e \) was calculated from contact depth of penetration \( h_e \) from the corrected recorded P-h datum fitting an area function related to the projected contact area to the projected contact depth as,

\[ A_e = C_0 \cdot (h_e^3) + C_1 \cdot h_e + C_2 \cdot h_e^{1.2} + \ldots + C_8 \cdot h_e^{0.128} \]

where \( C_0 \) up to \( C_8 \) are constants. Value of \( h_e \) can be estimated from the unloading cycle of the P-h curve as:

\[ h_e = h_{\text{max}} - \epsilon(P_{\text{max}}/S) \]

here \( \epsilon \) is a geometric constant ~0.75, \( S \) is stiffness, and \( h_{\text{max}} \) is maximum depth of penetration. \( S \) is the initial portion of the unloading curve, which was obtained by differentiating the load P with respect to the indentation depth \( h \) as,

\[ S = \frac{dP}{dh} = B \cdot m \cdot (h - h_e)^{(m-1)} \]

where \( B \) and \( m \) are compensation parameters and \( h_e \) is the residual indentation depth reached during the tests. Using these equations hardness \( H \) was calculated as a function of the indentation depth \( h \) [41-43]. The term \( dP/dh \) is calculated at \( h = h_{\text{max}} \). 52 mm long rods with 4 mm diameter along the grips, 3.5 mm along the gauge length and a length of 17.5 mm along gauge length were used to measure the tensile test using an INSTRON 8562 testing facility at room temperature with a strain rate of \( 1 \times 10^{-4} \) s\(^{-1}\). The strain during the tensile test was measured directly on the specimen using a Field laser-extensometer. All the tensile rods were build perpendicular to the substrate and the tests were carried out in the same manner (along the built direction).

2.1. Microstructure

A detailed microstructural and structural characterization evaluation of the as-prepared Al-12Si SLM specimens have been reported elsewhere [14,44] and only necessary details are discussed here in the context of the manuscript. Fig. 1 display the microstructure (both OM and SEM images) of the SLM samples prepared by SLM. It can be observed from the optical images (Fig. 1(a and b)) that the microstructure is not uniform. Typical impression of hatches were present (Fig. 1(a)) along with differences in the microstructure along the hatch overlaps (a relatively coarse microstructure is observed) and the core of the hatches with fine microstructure (Fig. 1(b)). In addition, the high resolution back scattered image show the presence of cellular microstructure. The thickness of the cell walls were observed to be ~200 nm and the size of the cells range between 500 and 1000 nm, which is in agreement with the previously published reports [10,14,35,44]. The EDX measurement (not shown here) confirms that the cell boundaries are rich in Si and the core of the cell are made of Al. The Si phase is present in the form of a continuous skeletal network [14,16]. The size of the cells were found to be bigger along the hatch overlaps (Fig. 1(b)), which are ~800–1000 nm and the core of the tracks contain cell with size ranging between 500 and 700 nm. This clearly shows the inhomogeneity in the microstructure and it may significantly affect the mechanical properties.

2.2. Mechanical properties

Fig. 2 show the mechanical properties (nano- and micro-indentation and tensile stress-strain curves) and the corresponding microstructural images of the Al-12Si SLM prepared samples. Fig. 2(a) shows a schematic of a typical nano-indentation curve of load (P) vs. depth (h) data obtained from the room temperature nano-indentation test results. The curve shows the presence of typical three segments: loading (which includes both elastic and plastic components), holding at maximum load and unloading (includes only elastic component) and is smooth without any evidence of pop-in effects [45]. The absence of displacement bursts or pop-in events within the P-h curve suggests that there is no elastic to plastic transition taking place with the Al-12Si SLM material. Further, it may be observed from the P-h plot presented in Fig. 2(a) that the maximum depth of penetration was \( h_{\text{max}} \) while the final depth of penetration was reduced to \( h_e \), as the sample elastically recovers, once the load is released. This marks the presence of the residual impression in the nano-indentated Al-12Si SLM sample [45]. In addition, no evidences of crack scan can be found from the P-h curve, since they are smooth and continuous. The SEM image of the Al-12Si SLM sample after nano-indentation is shown in Fig. 2(b). All the indents were perfectly positioned and presence of any defects like cracks or fracture surrounding the indents were completely absent corroborating the P-h curve. However, high magnification images (not shown here) show that the size of the
indents were not the same, suggesting the hardness values are not same throughout the material.

In order to map the local properties across the melt pools of the laser tracks or in the layers, nano-indents were spaced every 10 μm within a row and 15 μm between rows. The spatial resolution is selected in such a way that it covers both the hatch cores and the overlaps at least twice. Total 300 indents were performed across the entire surface of the Al-12Si SLM specimen and the resultant hardness contour map is shown in Fig. 2(c). The average hardness value from the nano-indentation measurements were observed to be $1.85 \pm 0.5$ GPa, with a minimum hardness value of $0.95$ GPa (Al-rich phase/pure Al) and a maximum hardness value of 8 GPa (corresponding to nearly pure Si phase) [46]. Fig. 2(c) clearly shows that the hardness in the Al-12Si SLM sample vary as a function of distance and never remains a constant. Carefully analyzing the contour plot (Fig. 2(c)) with the SEM image (Fig. 2(b)), it has been observed that for every $100-125$ μm in distance, the hardness of the Al-12Si SLM material increases, suggesting the presence of hatch overlaps, where concentration of Si is double than the core of the hatches [10,14]. Moving to the core of the hatches from the overlaps, there is a drop in hardness because of considerable less Si content, which can be evident from the contour plot (Fig. 2(c)). Hence, the hardness values from the nano-indentation test suggests that the local mechanical properties vary from hatch overlaps to the core of the hatches.

The Vickers microhardness test was carried out over 10 rows with at least 10 indents in each row with a spacing of 25 μm between each indents and between each row. The hardness data along each row are plotted and is shown in Fig. 2(d) and the corresponding SEM image with the indents are shown in Fig. 2(e). It can be observed from Fig. 2(d) that a least hardness of $125$ HV ($1.25$ GPa) and a maximum hardness of $175$ HV ($1.75$ GPa) was observed with a mean hardness ($1.5$ GPa). Even though the microhardness values do not fit perfectly with the nano-indentation results (due to size effects), the following conclusions can be drawn: (1) the hardness of the Al-12Si SLM sample do not remain constant and varies as a function of distance (hatch overlaps and hatch cores) and (2) a minimum hardness is observed for Al-rich regions (hatch cores) and the maximum hardness is observed along the Si-rich regions (hatch overlaps). Fig. 2(f) shows...
the true stress–true strain curves of Al-12Si sample tested at ambient conditions. Six samples were tested and the all the six samples are shown in Fig. 2[f]. It can be observed from the true stress–true strain plots that the Al-12Si SLM samples show nearly uniform properties with a yield strength of ~240 MPa, ultimate strength of ~385 MPa and a ductility of ~1.8–2.5%. The results are in agreement with the previously published reports [10,14,35,44]. They suggest that the samples are reproducible within experimental errors. Combining the hardness data and the tensile test results, it may be safely concluded that the Al-12Si SLM samples show repeatable bulk mechanical properties, even though variations in the local mechanical properties are observed due to anisotropy in the microstructure between hatch overlaps and the core of the hatches.

3. Summary
Al-12Si samples are fabricated using selective laser melting (SLM) process, which shows the presence of a cellular microstructure with Al phase along the core of the cells and Si phase along the cell boundaries in the form of a continuous skeletal network. The microstructure is anisotropic with coarse microstructure observed along the hatch overlaps and a fine microstructure along the core of the hatches. Both the nano-indentation (average hardness ~1.85 GPa) and micro-indentation experiments (average hardness ~1.5 GPa) (even though the average hardness values vary within experimental limits due to size effect) show the presence of variation in the local mechanical properties, due to the local variations in the microstructure as a function of position (hatch core/hatch overlaps). However, the tensile test data show repeatable mechanical properties (within experimental errors), giving evidence that the variations in the local mechanical properties do not have a significant influence on the bulk/macroscopic mechanical properties in the SLM fabricated Al-12Si samples.

Acknowledgements
Authors would like to thanks Dr. S. Scudino (IFW-Dresden, Germany) and Dr. F. Spieckermann (MontanUniversität Leoben, Austria) for stimulating discussion and assistance in carrying out some of the experiments.

References
[29] Maity T, Das J. Microstructure and size effect in ultrafine (Ti0.705Fe0.295)3Ti1-xSnx (0 ≤ x ≤ 4 at.3%) composites. J Alloys Compd 2014;585:54–62.