Preliminary microstructural investigation of Mg cubes produced by SLM

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INTRODUCTION: The ability to fabricate customized and resorbable metallic implants provides significant improvements in osteosynthesis. We try to combine the benefits of resorbable magnesium alloys with the versatility of the selective laser melting (SLM) process [1]. Preliminary metallographic investigations, SEM and XPS measurements of the starting raw material (spherical Mg powder, d_{50}-value 57 µm) and of the created objects indicate that a surface oxide layer limits the fusion of the powder during the SLM process.

METHODS: 3D Mg objects (Fig. 2a) were built in a SLM Realizer 100 machine (MCP Realizer, Germany). A 100 W continuous wave Ytterbium-fibre laser with a wavelength between 1068 and 1095 nm is used to melt the magnesium powder on a Mg substrate. The chemical surface composition of the Mg powder (AZ91, SFM, Switzerland) was analysed by XPS (PHI 5600, Physical Electronics, USA). For depth profiling, the surface of the embedded powder was repeatedly argon sputtered (600 s, 3 kV, 1.6 µA, $2 \cdot 10^{-8}$ torr) and analysed by XPS.

To reveal the microstructure, polished samples were etched with a 4% Nital solution for 1 min. Comparative images of the polished Mg structures before and after etching were taken with an SEM (TM-3030Plus, Hitachi, Japan). The porosity of the samples was visualized and optically quantified by light microscopy (BX61, Olympus, Japan).

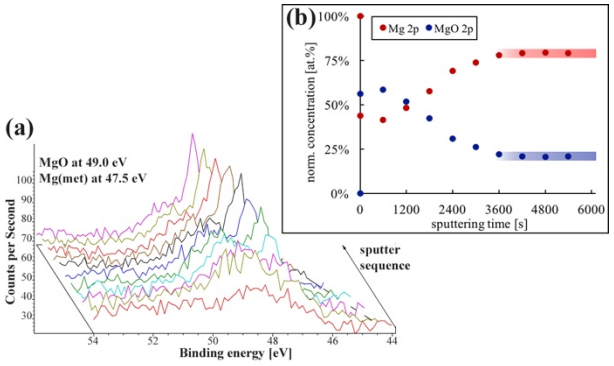

Fig. 1: XPS depth profiling. (a) Stacked Mg 2p spectra of Mg powder during Ar sputtering and (b) depth profiles of the metallic Mg and the MgOxide peaks.

RESULTS: The Mg2p and MgO2p peaks of the powder saturate after 3600 s sputter time (see Fig. 1a and 1b). With the sputter rate of 0.43 pm/s, the thickness of the surface oxide layer was determined as 1.5 nm.

Fabrication of Mg cubes by SLM was successfully achieved within a wide range of process parameters. Although the cubes show a solid appearance (Fig. 2a), a significant inner porosity was found after metallographic preparation (Fig. 2b). Polished and etched cross sections exhibit shell-like structures with inner skeletons (Fig. 2c).

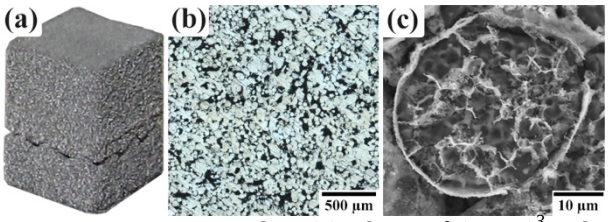

Fig. 2: Mg SLM samples. (a) Photo of (5 mm)^3-cube with subjacent support structure, (b) light microscopy picture of the cross section, (c) after Nital etching, residual shells from the oxide layer were discovered in SEM analysis.

DISCUSSION & CONCLUSIONS: The measured thickness of the Mg oxide layer is in line with literature [2,3]. Melting of the powder particles' oxide layer is found to be a major challenge in the SLM-treatment of Mg [4]. The high porosity of the investigated 3D-structures may correlate with limited fusion of the powder caused by the particles' oxide layer. The shell-like etching residues are supposed to consist of the MgO layer, while the skeletal structures are assigned to the intermetallic phase $Mg_{17}Al_{12}$.

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