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Investigation of the phase transition in SLM fabricated NiTi samples

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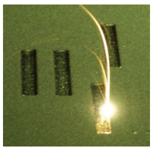
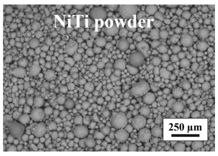
INTRODUCTION

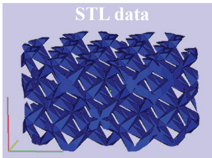


NiTi - an FDA-approved material - belongs to the shape memory alloys (SMA) which exhibit superelasticity, shape memory effects and damping properties. As the effects occur in the physiological temperature range, NiTi is perfectly suited for medical implants and instruments. By the additive manufacturing technique of selective laser melting (SLM), we fabricate complex-shaped NiTi scaffolds. Furthermore, we use the SLM process to create NiTi parts with different phase transition temperatures[2], which have been characterized by X-ray diffraction to identify the crystallographic phases at different temperatures. Based on properties tailored within the SLM fabrication process, we are going to realize porous implants, which should exhibit improved interactions with the surrounding biosystem.

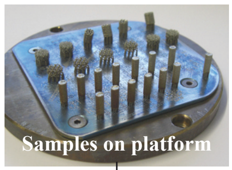
SAMPLE FABRICATION BY SLM

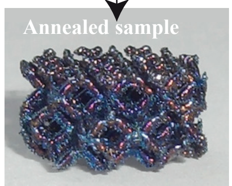
NiTi-specimens were manufactured by SLM from NiTi-powder (MEMRY GmbH, Weil am Rhein, Germany) using the SLM Realizer 100 (SLM-Solutions, Lübeck, Germany). Phase transition temperatures were tailored during the SLM fabrication process with varied energy densities and solution annealing at 800 °C. All processing steps were carried out under Ar atmosphere to protect the material from oxidation. The samples used in the presented investigations have austenite peak temperatures A_p of -3 °C (Sample 1), 50 °C (Sample 2) and 20 °C (Sample 3). Differential scanning calorimetry (DSC) and X-ray diffraction (XRD) measurements were accomplished at NiTi-powder and the specimens. XRD investigations were conducted at 34 °C.





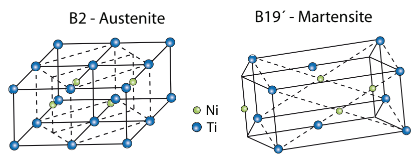
Processing parameters



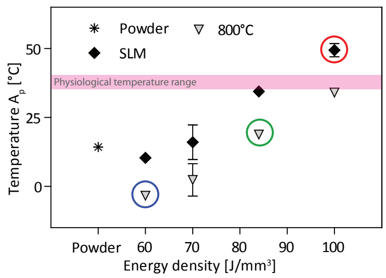


PHASE TRANSITION

The shape memory effect and the superelasticity are caused by a reversible martensitic phase transition. Depending on sample temperature or stress, either the high temperature austenite phase (B2) or the low temperature martensite phase (B19´) is stable. The change between the two crystalline phases doesn't involve atomic diffusion but a shearing of the crystal lattice. In certain cases, the phase transformation involves a third NiTi-phase, the so called R-phase. Here, a two step transformation takes place (B2 ↔ R ↔ B19´).[4]



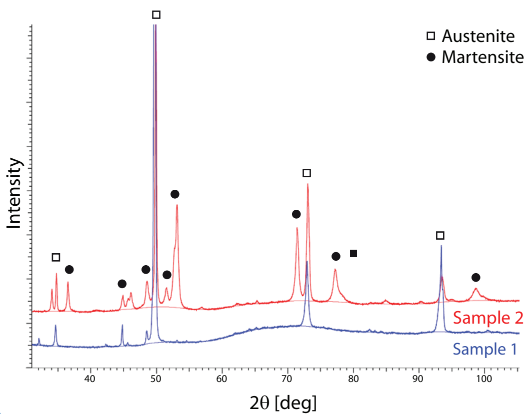
Depending on our processing route, samples with different phase transition temperatures were created.

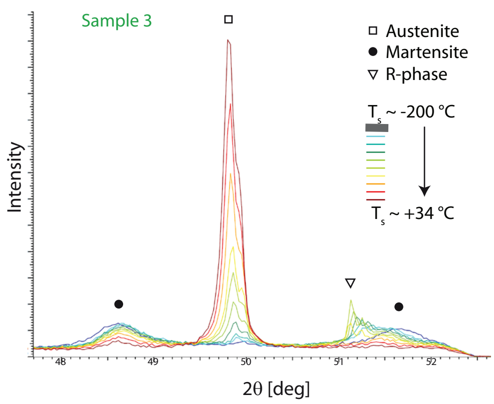


X-RAY DIFFRACTION

As seen below, the peaks of Sample 1 mainly relate to austenite with a cubic crystal lattice. Since its austenite peak temperature A_p lies below room temperature, the material is in austenitic phase. For Sample 2, whose A_p lies above room temperature, mainly martensite phase is expected. The spectrum, however, shows that both phases - martensite and austenite - are in coexistence. As seen from DSC investigations, the B19´ → B2 transition in Sample 2 starts already at 26 °C. However, the temperature within our X-ray diffractometer lies above room temperature at ~ 34 °C. Reason for the austenite phase is therefore an already started phase transition.

To dynamically investigate the phase transformation, Sample 3 was investigated at changing temperature. Its temperature increased steadily from around -200 °C (LN_2) to around 34 °C while various spectra where recorded. The scanned 2θ range (47.5° - 52.5°) covers austenite and martensite peaks. The R-phase was found to be involved in the phase transition, as we find a peak associated with a rhomboedrical lattice appearing and vanishing during warming of the sample. This indicates a two step transformation via the R-phase (B19` → R → B2) in our SLM specimens.





CONCLUSION & ACKNOWLEDGEMENT

By XRD, different crystallographic structures were identified in our SLM specimens, which have been produced with varied energy densities and subsequent solution annealing. Besides the austenite - martensite transition, we find a two step transformation route via the R-phase, which only exists in a narrow temperature range and does not appear in the DSC-curves. The SLM process allows the production and simultaneously tailoring properties of complex-shaped NiTi specimens in only one fabrication step. Furthermore, anisotropic properties can be achieved. Based on these findings we will realize NiTi-scaffolds in which several effects can be exploited for an advanced performance.

We sincerely thank the Co. MEMRY GmbH for powder- and knowledge-supply. Furthermore, the multi-disciplinary team gratefully acknowledges the financial support of the Swiss National Science Foundation within the program NRP 62 'Smart Materials'.

References: [1] ASTM International, F 2063-05. [2] Bormann T. et al. (2010), Proc. SPIE 7804: 78041. [3] Allafi J.K. (2002), Dissertation. [4] Otsuka K. et al. (2005), Prog Mat Sci 50: 511-678.