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Texture and anisotropy in selective laser melting of NiTi alloy

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ABSTRACT

Selective laser melting (SLM) is used to manufacture dense NiTi parts. The microstructure and texture are assessed (before and after annealing followed by furnace cooling) and linked to the compression behaviour and shape memory response. It is shown that SLM strongly orients the fine austenite subgrains towards the building direction. This texture induces the highest spring back along the building (vertical) direction and the lowest along the horizontal direction after compression. The compressive stiffness, on the other hand, is the highest along horizontal direction and the lowest in vertical direction. The internal stresses due to SLM processing are another factor that may induce large martensite plates, decreasing the spring back. Although post-annealing (followed by furnace cooling) annihilates these large SLM stress-induced martensite plates, it is unsuccessful to achieve completely isotropic properties. The furnace cooling after annealing may even segregate austenite and martensite within SLM solidified tracks, causing a mixed shape memory response.

1. Introduction

NiTi alloys are widely used in medical and non-medical applications owing to their shape memory response [1–3]. The shape memory response is the deformation recovery of the material upon heating to above specific temperatures after being deformed at lower temperatures. The shape recovery originates from a reversible martensitic transformation between the low temperature phase (martensite) and the high temperature phase (austenite). In fact, the low temperature martensite deforms through self-accommodation of twin variants with gliding interfaces. Subsequent heating transforms the martensite (monoclinic with low symmetry) to austenite (bcc with high symmetry) and recovers the original shape (called thermal memory) [4,5]. In a contrary scenario when the austenite is stressed at a specific temperature, it transforms to martensite. Since the martensite is unstable without stress at those temperatures, the martensite transforms back to austenite upon unloading, recovering the deformation. This results in a very large elastic response and spring back [6]. Obviously, these behaviours depend on the microstructural characteristics, including the morphology and texture of the material (even in polycrystals) which manipulates the transformation strain and symmetry of martensite during the transformation [5,7,8]. For example, Gall and Sehitoglu [7] have shown that the stress can be typically higher in a uniaxial compression test than that of tensile loading for a textured material (depending on the texture orientation), in contrast to an almost comparable stress–strain behaviour in compression and tension for a randomly oriented NiTi polycrystal.

Due to the wide applications of NiTi shape memory alloys, they are now being developed (either as bulk [9–11] or porous materials [12,13]) by advanced manufacturing processes such as selective laser melting (SLM). SLM is a powder based additive manufacturing method in which a complex three-dimensional part is built by melting and solidifying the required geometry, layer-by-layer on top of each other [14,15]. The complex geometry and free-form fabrication offered by SLM is an excellent opportunity to even further expand the applications of NiTi alloys into very advanced/complicated parts (e.g. biomedical scaffolds [13], actuators [1], surgical devices [16], etc.).

After manufacturing dense NiTi components using SLM, the main challenge is to distinguish the influence of different process parameters altering the shape memory properties of the NiTi products. For example, Bormann et al. [10] have observed that higher laser energy densities increase the martensitic transformation temperatures, or Dadbakhsh et al. [11] have reported that higher scanning speeds (adjusted to higher laser powers) decrease the transformation temperatures and stabilise the austenite. Despite these efforts, the influence of the strong SLM texture (originating from the directional cooling and layer-by-layer nature of SLM) [17,18] is still an unknown factor for NiTi. Since this unknown factor can potentially affect the properties and hence the final application of the products [19–21], further investigations are required to explore the shape memory and mechanical anisotropy

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after SLM of NiTi.

This work aims at elucidating the SLM induced texture of bulk NiTi alloys. The results are linked to the anisotropic compression behaviour and shape memory response. High temperature post-annealing followed by furnace cooling is also employed to clarify the ultimate influence of temperature on the texture and shape memory response. The findings are to provide a basic materials knowledge required for NiTi development using the SLM process.

2. Material and experiments

2.1. SLM process

Plasma atomised NiTi powder (nominal Ni content of 50.2 at%) was provided from Raymor Industries Inc., Canada. The particle size ranged from 25 μm to 45 μm (Fig. 1). The appropriate size in conjunction with the smooth surface and spherical morphology of the powder particles allowed a well-packed and uniform layer deposition. An in-house SLM machine (300 W Yb:YAG fibre laser with a beam diameter of about 80 μm) was used to manufacture multilayer samples under argon atmosphere. Successful parts ($6 \times 6 \times 12\ mm^3$ with densities over 99%) were manufactured using a layer thickness of 30 μm, a laser power of 250 W, a scanning speed of 1100 mm/s, and hatching space of 60 μm. The parts were oriented horizontal (0°), at an angle (45)°, and vertical (90°) on the SLM baseplate (Fig. 2). Each layer was scanned once. A bi-directional scanning was carried out and a 90° rotation was applied between layers (Fig. 2a).

2.2. Characterisation

In addition to as-built products, analysis was also carried out after full annealing at 830 °C for 25 min followed by furnace cooling under argon atmosphere. The transformation behaviour of both as-built and annealed samples was characterised by DSC with a cooling/heating rate of 10 °C min^{-1} from −80 °C to +130 °C.

To investigate the mechanical behaviour and shape memory response, the samples were compressed using a 250 kN INSTRON universal testing machine with a crosshead speed of 0.2 mm/min at room temperature. The top and bottom surfaces were machined beforehand and zinc stearate was used as lubricant. After 4% and 8% compression, the samples were stored in a cold box (filled with ice) until they were heated from RT to 130 °C using a dilatometer (DIL 402 C/7 Netzsch) to record the changes in length during heating. The dilatometry was carried out with a 5 °C min^{-1} heating rate under helium atmosphere.

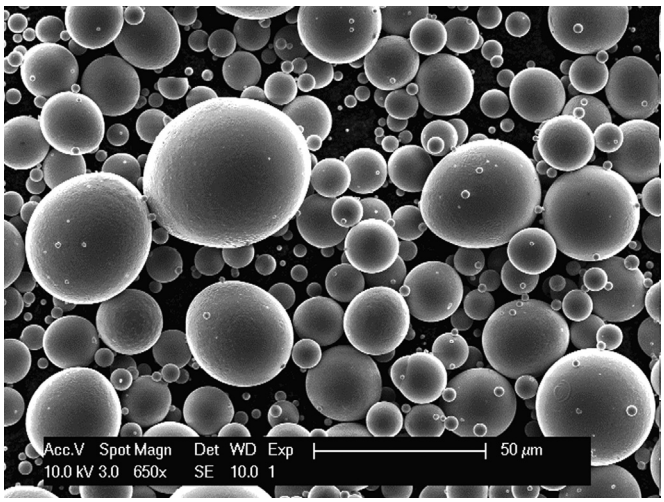

Fig. 1. SEM picture of plasma atomised NiTi powder.

Phase identification was carried out using a Siemens D500 X-ray diffractometer (XRD) with coupled Theta/2Theta scan type and Cu-Kα1 radiation (wavelength: 0.15418 nm) operated at 40 kV and 40 mA. XRD was also used to evaluate the texture on side cross-sections parallel to the building direction. The pole figures of (110), (200), (211) and (310) reflections were measured and corrected in comparison with as-received NiTi powder for cubic structure. Also, the pole figures of (002), (111), (012) and (003) reflections were measured and corrected in comparison with annealed and furnace cooled NiTi powder for monoclinic structure. The rotation of the sample around the intersection line of the sample surface with the plane of the incident and diffracted X-rays was recorded in steps of 5° leading to circles and contours from the diffracted beam. The MTM–FHM texture processing software [22] and MTEX Software Toolbox [23] were used to calculate the pole figures, texture indices and normalised texture differences.

The sample cross-sections were viewed using an Axiocam Leica optical microscope after chemical etching with a solution composed of 5 ml HF, 25 ml HNO_3, 50 ml H_2O at RT. EBSD was performed using a FEI XL30 scanning electron microscope equipped with a TSL orientation imaging system.

3. Results

3.1. Phase analysis

XRD results, shown in Fig. 3, identify the phases present after SLM and also after annealing treatment. As seen, the as-built parts mainly match with NiTi cubic (austenitic) B2 peaks. However, it also contains some minor/weak peaks belonging to the monoclinic (martensitic) B19′ structure. Subsequent annealing significantly intensifies the monoclinic peaks. The presence of very small amount of precipitates (such as $NiTi_2$,Ti_4Ni_2O, or Ni_4Ti_3) could also be sensible after furnace cooling from the annealing temperature, though the corresponding XRD peaks were very weak (and not labelled) due to very low fraction of these constituents (Fig. 3).

The differential scanning calorimetry (DSC) curves, shown in Fig. 4, confirm the XRD results by showing a higher martensitic temperature for the samples after annealing compared to the as-SLM part. In fact, the as-built sample is mainly austenite (A) at room temperature, but annealing induces martensite in the parts. It is worth mentioning that the latent heat for all transformations (M→A and A→M) prior and after annealing was calculated to be 19–20 J/g. According to Khalil-Allafi and Amin-Ahmadi [24], and Frenzel et al. [25], this value indicates the presence of about 50.6–50.8 at% Ni in the alloy (being higher than the nominal recorded value of 50.2 at%). This means there is about 0.6–0.8 at% excessive Ni from the stoichiometric condition. This excess of Ni may promote Ni rich precipitates after heat treatment.

3.2. Compression performance and deformation recovery

Fig. 5 shows the compression behaviour of the NiTi samples; the samples were first unloaded after about 6% and then loaded again up to 18–20%. As seen, the orientation of the load with respect to the SLM building direction is very important. This is better clarified in Fig. 6 where the apparent stiffness (the resistance of material against loading, calculated from the initial slope of the unloading curve – see Fig. 5a) are plotted after about 4%, 6% and 8% of compressive strains. As seen, the resistance of the material against the compressive force is highest in the horizontal direction and lowest in the vertical direction.

Fig. 7 shows the deformation recovery of the parts depending