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Quantitative characterization of pore configuration in a porous Ni50.8 at.%-Ti SMA

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Abstract

A porous $Ni_{50.8}Ti_{49.2}$ shape memory alloy prepared by space holder assisted conventional sintering has been characterized via the micro-computed tomography (micro-CT). After proper image processing to remove the artifacts from the original reconstructed slices, the three-dimensional visualization, together with the size, shape and local structure of the pores have been obtained from the reconstructed volume. Primary pores with a volume fraction of 20.2% duplicate the size and morphology from the space holder particles and the space between the elemental nickel and titanium powders as raw materials, and segment the matrix into small volumes with an average diameter of around 220 μm and a distance of around 125 μm away from each other. This configuration in the porous $Ni_{50.8}Ti_{49.2}$ alloy can stall the propagation of martensite and lead to a gradual transformation.

Keywords: Porous Ni-Ti alloy; Pore configuration; Three dimensional reconstruction; Micro-computed tomography

1. Introduction

Porous Ni-Ti alloys have drawn increasing attention in the last decades following the dense Ni-Ti shape memory alloys (SMAs). It has been shown that porous Ni-Ti alloys inherit the outstanding properties from the dense ones, such as shape memory effect (SME) and superelasticity originating from the displacive martensitic transformation;

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and meanwhile, their unique features including light-weight, high strength, relatively low modulus, excellent bio-compatibility and pore structure enable them to be promising candidates of biomedical materials for hard-tissue replacement/repairing. So far, various methods based on powder metallurgy have been developed for the fabrication of porous Ni-Ti alloys, by which the pores with different characteristics can be introduced into the matrix of Ni-Ti alloys. It is found that the pore structure plays a crucial role in mechanical properties of the porous alloys. Moreover, the discontinuous geometry due to the porous structure will finally work on the diffusional transformation behavior (e.g., the precipitation process) in porous Ni-Ti alloys, which controls the diffusionless martensitic transformation and functional properties of the alloys. Therefore, it is important to obtain a comprehensive and quantitative understanding on the pore structure in porous Ni-Ti alloys [1]. Previous studies focused on revealing the three-dimensional (3D) visualization of the pores together with quantitative data such as the pore size, volume fraction (porosity) and number density in this type of material [2]. However, less attention has been paid to the morphology, especially the local structure of pores and the discontinuous matrix segmented by them.

In the present study, a porous $Ni_{50.8}Ti_{49.2}$ alloy was fabricated by the space holder assisted conventional sintering technique, which has been developed to produce porous alloys with controllable pore size and porosity. The as-prepared porous $Ni_{50.8}Ti_{49.2}$ alloy was then characterized via the micro computed tomography (micro-CT), in order to reveal the 3D pore configuration and the local matrix structure segmented by the pores. These results will offer a better understanding of the potential influence on the functional and mechanical properties brought by the pores in the porous Ni-Ti SMAs.

2. Experiments

Nickel and titanium powders (50 μm, 99.9% purity) with atomic ratio of 50.8%Ni to 49.2%Ti were blended for 24 h, followed by mixing with 5wt. % NH4HCO3 powder (200~300 μm, 99.99% purity) as the space holder for 3 h. Afterwards, the well-mixed powders were cold compacted into green samples with a geometry of 16×12 (diameter × height, mm^2) under a compressive stress of 100 MPa by a hydraulic presser. The green samples then underwent a gradient sintering in a quartz tube furnace under the protection of flowing argon (99.99% purity), with temperature elevated gradually from room temperature till 1273 K and held at the latter for 3 h. Afterwards, the as-received samples were subjected to a solid solution treatment at 1273 K for 10 h and an aging treatment at 773 K for 2 h, both under vacuum. All processes of sintering and heat treatments were followed by water quenching. The results of DSC tests and mechanical properties indicate that the heat treatments of solid solution and aging yield a typical B2-R-B19' transformation in the alloy and an compressive strength of 369 MPa at a strain level of 5%, which is much higher than that reported in previous studies [1,3], providing a high possibility for this alloy to be used in some applications in aeronautical and aerospace engineering.

One of the as-prepared samples was cut into a smaller cylinder of 6×10 (diameter × height, mm^2) to fit the micro-CT analysis. In this study, a micro-CT (ZKKS-MCT-Sharp-IV) was employed to acquire the 3D data of the porous $Ni_{50.8}Ti_{49.2}$ alloy. The sample was rotated from 0~360º with a rotation step of 0.45º, exposed by an X-ray of 80 kV, 0.75 mA. In this way, a sequence of 8-bit 480×500×740 grayscale raw slices with a voxel size of 15μm was generated by the Graphics Processing Unit (GPU) accelerated Feldkamp-Davis-Kress (FDK) algorithm, considering the compromise of the Field of View (FOV) and the resolution.

3. Results and discussion

3.1. Image processing

Fig. 1(a) shows one of the reconstructed slices of the porous $Ni_{50.8}Ti_{49.2}$ alloy obtained from micro-CT, dark parts within the alloy represent the pores. Besides typical defects of noise and gradient, subtle ring artifacts, which are a sequence of concentric rings due to miscalibrated or defective detector, can also be observed in the original image. These image defects prohibit further qualitative and quantitative analysis. In order to yield reliable 3D data, necessary image processing was performed by ImageJ© and Matlab© before 3D reconstruction in Amira©. A polar transformation in ImageJ© was applied to "unfold" the 2D images from the center of the concentric rings, by which the ring artifacts can be transferred into stripes. Afterwards, a Fast Fourier Transformation (FFT) filter in the frequency domain was applied to eliminate the stripes in the polarized images, followed by transferring back to the real space with an inverse FFT. Then the filtered polarized images were "bent" back to the Cartesian coordinates [4]. After the above processes presented in Fig. 1(b) – (e), the 2D images were subsequently optimized with a band-pass filter to remove the noise and background, followed by a threshold process to segment into binary ones in a selected region of interest [5,6], of which one example is shown in Fig. 1(f). Finally, a 3D volume of $3.75 \times 3.75 \times 6$ mm^3 was reconstructed in Amira© from the 2D binary image sequence (see Fig. 2(a)).

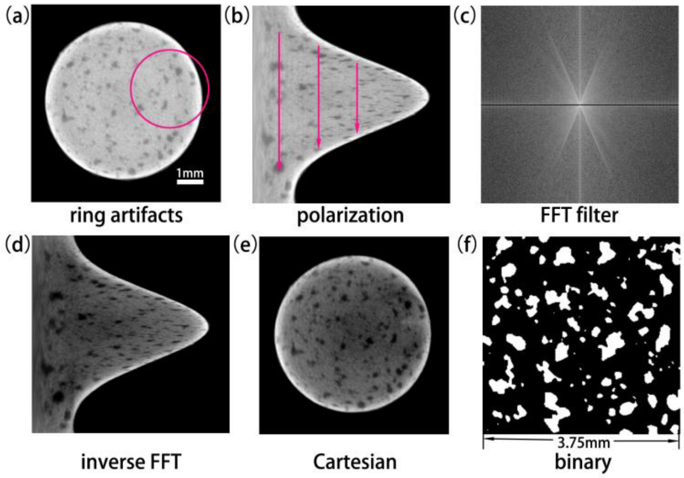

Fig. 1. One example of the original reconstructed slices of the porous $Ni_{50.8}Ti_{49.2}$ alloy obtained from micro-CT showing subtle ring artifacts marked by the pink circle (a); Polarized image with stripe artifacts indicated by the arrows (b); FFT filter to eliminate stripe artifacts in the frequency domain (c); Polarized image transferred back to the real space with an inverse FFT following FFT filter, showing no stripe artifacts (d); Cartesian image after removing ring artifact (e); One of the binary images in the region of interest after image optimization and segmentation (f).

3.2. Qualitative and quantitative analysis of 3D data

From the reconstructed 3D volume shown in Fig. 2(a), one can observe that pores of irregular shape distribute evenly in the porous $Ni_{50.8}Ti_{49.2}$ alloy, some of them tend to connect with each other. It should be noted that due to the limitation of resolution, only the primary pores normally with a size over 50 µm, which make major contribution to the functional and mechanical properties of porous Ni-Ti alloys, can be observed in this condition. This kind of pores originate from the natural space between the elemental nickel and titanium powders and occupation of space holder particles in the green sample, and thus inherit the size and shape from them. On the other hand, the secondary pores of much smaller size, which are induced by the residue of space holder particles after decomposition, non-metallic impurities in the raw materials, shrinkage due to sintering, and Kirkendall voids, can hardly be revealed in this case.

Based on voxel counting, parameters of volume ratio (f_v), size (V_p), number density (n) and spherecity (Ψ) of all pores in the 3D volume can be extracted. There are in total 2815 primary pores with an average V_p of 0.00604 ± 0.2338 mm^3 in the reconstructed region, yielding an n of 33.4 mm^{-3} and an f_v of 20.2%. Among all those pores exists an open one contacting all boundaries of the 3D volume, with a size of 12.404 mm^3 (much larger than the close ones of 0.0016 mm^3 in average), taking a volume fraction of 72.9% over all primary pores. This result confirms a well connection of the pores as observed in the 3D visualization, which may be attributed to the assistance of the secondary pores around the primary ones. This can also explain the origination of the extremely large standard deviation of the average V_p. All primary pores yield an average Ψ of 0.84 ± 0.12, corresponding to a large amount of the closed pores. This can be explained by the fact that the closed pores duplicate the irregular but near equiaxial shape from the space between Ni and Ti powders, as well as the space holder of NH_4HCO_3 particles in the green sample.

To better understand the spatial configuration of the pores and the segmented matrix in the porous $Ni_{50.08}Ti_{49.2}$ alloy, a "local thickness" algorithm [7] was applied to reveal the local structure of both the pores and matrix. The thickness of every local point in the structure was calculated, which is defined as the diameter of the maximal sphere fitting in the object and containing the corresponding point. The 3D local thickness maps of the pores and matrix in the reconstructed region along with their histograms are shown in Fig. 2(b) and (c) respectively. It can be found that the local thickness of the pores, which can be understood as the diameter of them, ranges from 30~375 μm, leading to an average of 125±57 μm. Meanwhile, the local thickness, or the diameter of the matrix, falls in the range of 30~555μm, with an average of 220±70 μm. This implies that the primary pores in the porous alloy segment the matrix into small volumes with an average diameter of around 220 μm and separate them with a distance of around 125μm. These small volumes of the matrix in such a size still allow the martensite to nucleate and grow, but the distance between them can stall the propagation of the martensite and lead to gradual transformation [1].

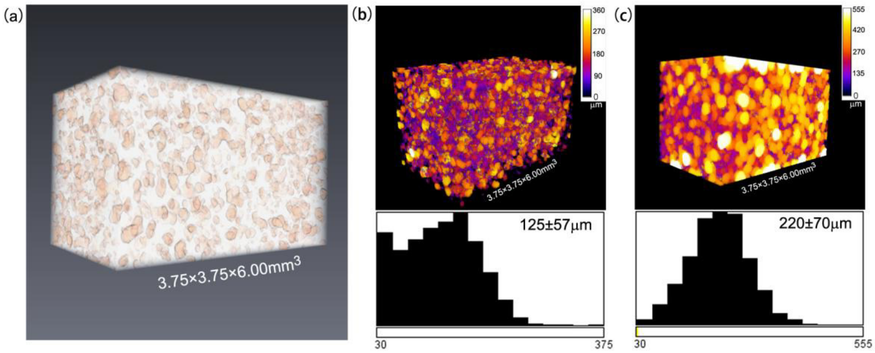

Fig. 2. 3D visualization of the porous $Ni_{50.8}Ti_{49.2}$ alloy in the selected region of interest (a) and the local thickness maps and corresponding histograms of the pores (b) and the matrix (c).

4. Conclusion

The 3D configuration of the pores and matrix in a porous $Ni_{50.8}Ti_{49.2}$ alloy has been characterized by a micro-CT analysis. Polarization method combined with the FFT filter can be used efficiently to remove the ring artifacts from the original slices obtained from micro-CT. A large amount of primary pores can be observed in the reconstructed region, which inherit the size and shape from the space holder particles and the natural space between elemental nickel and titanium powders, and well connect to form large open ones. The matrix of porous alloy is segmented by the pores into small volumes with an average diameter of around 220 μm and a distance of around 125 μm away from each other. This configuration in 3D can stall the martensite propagation during the transformation and thus yield a gradual transformation process.

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