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Quantitative tomography of pure magnetic-induced effects on metallics during microwave sintering

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ABSTRACT

Directional mass transport phenomena that differ from traditional sintering was found during microwave sintering of Ni–Ti mixed powders, and the underlying magnetic-related mechanisms were analyzed, which benefitted from a newly developed experiment setup that combining a single-model microwave cavity with the synchrotron radiation computed tomography (SR-CT) system. The electric and magnetic fields (H-fields) of microwave can be separated and their direction and amplitude become quantifiable and definitive by using the single-mode microwave cavity, and the microstructure evolution of matter in separate H-field can be online observed by the SR-CT technique. The experimental results showed the presence of special mass transport from Ti particle to Ni particle. It may be caused by different magnetic energy loss in Ni and Ti particles due to their different dielectric properties. The mass transport from Ti to Ni was found to have unequal rates in different directions, which may be caused by the polarization direction of H-field. This study may provide some inspiration for fabricating new metallic materials with oriented microstructure and property by using microwave H-field. It also proves the effectiveness of the single-mode microwave cavity SR-CT experimental setup and provides a new way for in-depth investigation on microwave interaction with matter.

1. Introduction

The first successful microwave sintering of metal and alloy powders by Roy et al. [1] has opened up a new research fields that using microwave sintering to fabricate a number of advanced metallic materials with improved microstructure and functional properties [2–6]. Contrary to traditional theory on the subject of microwave interaction with matter, which assumed that heating in microwave field is caused by energy losses of electric field (E-field) alone, it was found that the magnetic field (H-field) may have more influence on metallic material than E-field [7–9]. For instance, Yoshikawa et al. [8] reported that metal powders were heated more effectively in H-field than in E-field.

However, the detailed mechanisms of H-field participating in the sintering process are still unclear, thus it has not been achieved to purposefully control the H-field during sintering and to obtain designed material properties. A widespread assumption is that the H-field could induce eddy current and generate heat in metals. But this assumption is too macroscopic and it ignores many microscopic details, especially the microstructure of metals. In fact, the H-field and the metal microstructure are coupled and can influence each other at micro-scale during sintering. The H-field could generate thermal effect and may have other direct effects [9–14] on metals (such as changing the reaction paths in sintering [9]). Under those coupling effects, the microstructure of metals would evolve complexly. In return, the evolved microstructure would influence the distribution of H-field, which would ultimately change the effects of H-field. As for alloy systems, the effects of H field may become more complex due to the non-uniform magnetic properties of heterogeneous metals. However, the studies on the subject of the interactions between H-field and metallic material is largely insufficient.

In order to reveal the interactions between H-field and metallics, the ideal method is to conduct quantitative experiment on the sintering process of metallic in pure microwave H-field. However, it is very difficult to carry out this kind of experimental research previously. The difficulties could be attributed to two aspects: the unquantifiable H-field parameters and the unsuitable microscopic observation technique. In most initial literature on microwave

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processing, the multi-mode microwave cavity was used [1,4–6], in which the E-field and H-field are mixed in space. The parameters of H-field (direction and amplitude) is time-varying and uncertain, so that it is impossible to investigate the effect of a single field in this cavity. Although later researchers began to use a single-mode microwave cavity to process materials [10,11,15], they still subject to experimental observation technique, and can only obtain the macroscopic sintering behavior of materials or analysis the microstructure change offline after microwave processing. They could not obtain the whole microstructure evolution information during sintering. But this information is very important for researching microwave-matter mechanisms. Therefore the current studies on interactions of H-field with metals remain lack of direct experimental investigation.

This paper presented an effective approach to investigate the mechanisms of H-field with metallic material by using a new experimental system, which is combination of a single-mode microwave cavity and an online synchrotron radiation computed tomography (SR-CT) technique. The E-field and H-field are separated and their direction and amplitude become quantifiable and definitive by using the single-mode microwave cavity, and the microstructure evolution of matter in separate H-field is online observed by the SR-CT technique. Benefit from this experimental setup, special phenomenon such as directional mass transport between Ni and Ti, was found during microwave sintering of Ni–Ti mixed powder, which were different from conventional sintering. The corresponding mechanisms were analyzed by combining the experimental results and microwave field simulation analysis. It was found that the H- field may induce different energy loss in Ni and Ti particles and motivate special mass transport between them. The direction of H-field may have an influence on the mass transport rates and induce the anisotropic mass transport. This study may provide inspiration for fabricating some high-performance metallic materials by using microwave H-field. It also proves the effectiveness of the single-mode microwave cavity SR-CT experimental setup and provides a new way for in-depth investigation on microwave interaction with matter.

2. In situ SR-CT experiment and special microstructure evolution phenomenon

2.1. Experimental procedure

The Ni–Ti mixed powder was selected as the experimental object because Ni and Ti have very distinct magnetic property, which may make them show different sintering behaviors in microwave fields. The average diameter of Ti and Ni powders is 50 μm. The powders were uniformly mixed at a composition of 51 at. % Ni and 49 at. % Ti and then pressed in a steel die under 50 MPa uniaxial pressure.

Microwave sintering was performed using a specially designed single-mode microwave resonance oscillation cavity system. The experimental setup (Fig. 1(a)) mainly consists of a 3 kW 2.45 GHz magnetron source, a rectangular resonator (222 mm long × 109 mm wide × 54 mm high), and a movable plunger. Microwave emitted by the magnetron source resonated as TE103 single mode in the cavity. The distribution of H-field is shown in Fig. 1(b). For convenience, we defined a three-dimensional Cartesian coordinate system, where the X-axis is parallel to microwave propagation. The electric vector of microwave oscillated along the Y-axis, and the magnetic vector oscillated in the ZOX plane. The intensity of the E-field and H-field was distributed a sinusoidal pattern along the X-axis. The distribution could be adjusted by tuning the movable plunger.

Prior to microwave sintering, the sample was placed in the resonator where the H- field intensity is maximum. During sintering, the microwave output power was gradually increased from 0 to 1600 W (Fig. 2). The sample was surrounded by an alumina-silicate box for microwave transparency and heat preservation. An infrared thermometer was placed above the sample for precise temperature measurements from 250 °C to 1800 °C. The temperature profile during sintering is also shown in Fig. 2. The microwave cavity was filled with argon gas to prevent sample oxidation.

On-line SR-CT observation was conducted on the BL13W1 beam line at the Shanghai Synchrotron Radiation Facility (SSRF) [16]. The photon energy of the X-ray beam was 38 keV. The spatial resolution used in the experiment was 0.33 μm/pixel. Tomography data of the sample were collected continuously during microwave sintering. Each tomography scan consisted of multiple exposures, each of 500 ms, collected at 1.5° angular steps over 180° rotation of the sample. The tomographic slices were reconstructed using an AFBP-TVM sparse algorithm [17–19]. The slices were stacked into 3D images and visualized in Volume Graphics (VG) software. Fig. 3 shows the full-field microstructure evolution of the Ni–Ti mixed powder during microwave sintering. The grayscale range of a reconstructed slice was 0–255. The grayscale range corresponding to Ni was 195–255 and the grayscale range corresponding to Ti (together with Ni–Ti intermetallics) was 59–194. Therefore different materials in the sample could be distinguished according to their gray levels. This image segmentation method was proved to be reliable in this experiment, because the relative proportion of Ni regions and Ti regions after segmentation was consistent with the mixing proportion of the sample before sintering. In Fig. 3, different materials were colored for illustration. The orange regions represent the Ni particles and the gray regions represent the Ti particles (together with Ni–Ti intermetallics). The red arrows in Fig. 3(a) indicate the schematic of the instantaneous H-field.

2.2. Special phenomena during sintering of Ni–Ti mixed powder in H-field

Based on 3D images in Fig. 3, interesting phenomena were observed during microwave sintering of the Ni–Ti mixed powder. The first special phenomenon was the gradually decreased volume of the yellow regions (Ni) and increased volume of the gray regions (Ti and intermetallic compounds).

This special phenomenon differed from the microstructural evolution during conventional sintering. Fig. 4(a) and (b) show the vertical sections of the sample cut along the yellow planes in Fig. 3. As a comparison, Fig. 4(g) and (h) demonstrated the SEM images of conventional sintered Ni–Ti alloy (reported by Whitney et al. [20]). The Ni particles (white) were eroded by its neighboring Ti particles (gray) during microwave sintering, in contrast to the diminished Ti cores (black regions) and increased Ni (white regions) during conventional sintering.

The interesting phenomenon was further investigated. The phase composition of the sample under different sintering durations was characterized by XRD technique to determine the phase transition in the mixed powder because of the close relationship between mass transport and phase transition. The tested samples were microwave sintered by the same heating profile as that in the SR-CT experiment; the maximum sintering temperatures were controlled at 700 °C, 900 °C and 1100 °C (shown as #2 to #4 in Fig. 2). As shown in Fig. 5(a), the XRD patterns only contain peaks of Ni, Ti, and intermetallic compounds, and no peaks of oxide were found in the raw and sintered materials. This finding suggests that the protective atmosphere prevented the oxidation of samples during microwave sintering. Furthermore, the XRD results demonstrated the phase evolution during microwave sintering that corresponded to the results of SR-CT experiment. At 700 °C, the