

# Microstructure and texture evolution of ultra-thin TiNi hot-rolled sheets studied by automated EBSD

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## Abstract

Electron backscattering diffraction (EBSD) investigations were carried out on three ultra-thin TiNi rolling sheet samples with thickness of 0.15, 0.2 and 0.3 mm, respectively. The microstructure and texture evolution were investigated as a function of the sheet thickness. The automated EBSD system allows integrating the grain boundary, grain size and the preferred orientation information to be revealed simultaneously. By ODF analysis, the preferred orientations are revealed as  $\{111\} \langle 0\bar{1}1 \rangle$ ,  $\{223\} \langle 0\bar{2}1 \rangle$ , and  $\{332\} \langle 1\bar{1}0 \rangle$  in the three TiNi sheets. The  $\{111\} \langle 0\bar{1}1 \rangle$  component evolves stronger with reducing the thickness of the rolling sheet.

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**Keywords:** EBSD; TiNi; Grain boundary; Preferred orientation

## 1. Introduction

TiNi shape memory alloys are well known for their shape memory effect (SME) and super-elasticity. The SME and super-elasticity have been employed in many fields such as: aerospace, military and medical devices. The super-elasticity of TiNi alloys has been proved to be a function of texture, which is closely related to the thermal–mechanical history and the status of the materials. Texture features of rolling sheets, drawing wires and tubes are very different [1–4], the effects of testing temperature, alloy composition and thermal–mechanical treatments have been studied extensively [5–9]. However when the thickness of the rolling sheets becomes ultra-thin, for example less than 0.5 mm, the effect of thickness to the texture must be considered. In fact, the recoverable strain, the super-elasticity and some other mechanical features are not only

a function of texture, but also the grain size and sub-structures such as the low-angle grain boundary distribution, density, etc. Only very limited investigations integrated these information in one study. This was partly because of the difficulties of preparing the samples and also the limited ability of the applied technical means. In this investigation, an automated electron back-scattered diffraction (EBSD) system with a SEM was employed to study the texture and microstructure evolution features of a group of TiNi rolling sheet samples as a function of the sheet thickness. The integrated information of low-angle grain boundaries, preferred orientation distribution and grain size evolution have been studied as the function of the rolling sheet thickness.

## 2. Experimental details

Three near equal-atomic TiNi hot-rolled sheet specimens with thickness of 0.15, 0.2 and 0.3 mm were prepared by GEE Corporation, China. All of these specimens possess super-elasticity at room temperature. The EBSD data collection was conducted on a JEOL 6500 scanning

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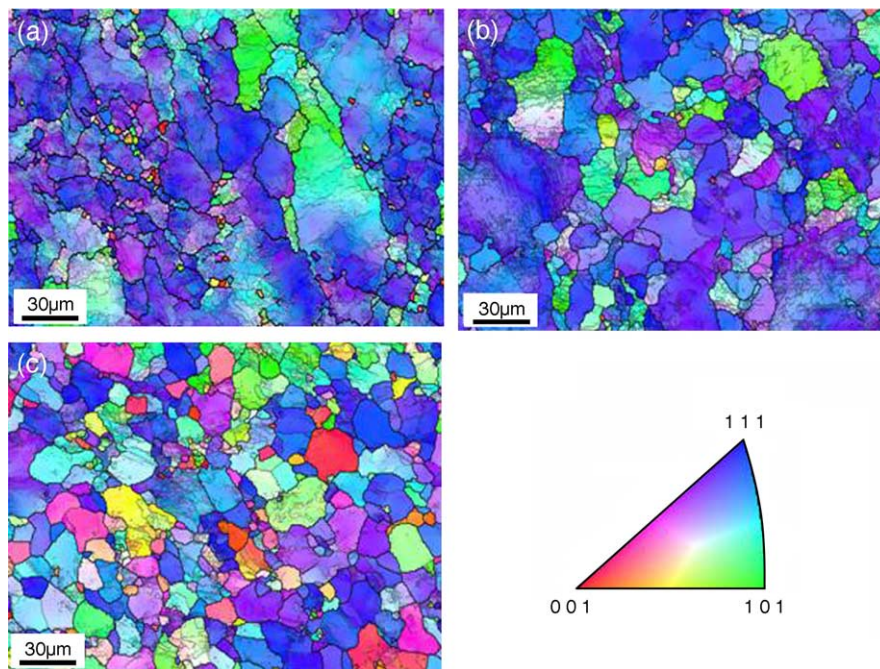


Fig. 1. Tri-color inverse pole figure map overlapped with grain boundaries of three TiNi sheets, (a) 0.15, (b) 0.2, and (c) 0.3 mm.

electron microscope (SEM) with a field emission gun (FEG). The samples were mechanically polished followed by an electrolyte polishing to the final quality for EBSD study. The electrolyte solution is 25%  $\text{HNO}_3$  with 75% methanol. The electro-polish was conducted at  $-30^\circ\text{C}$  and 20 V. The automatic EBSD data were collected with a commercial TSL system and analyzed by the software. The collected EBSD data were filtered by confidence index value of 0.2.

### 3. Results and discussion

#### 3.1. The low-angle grain boundary and grain size evolution through the rolling process

LAGB intensity and distribution is an important indicator of the microstructures of a material. It implies the dislocation density and distribution, the sub-grains, and dislocation–cell evolution information from a deformed matrix. In the EBSD analysis, the LAGB is defined by the algorithm of nearest-neighbor disorientation angle. The length fraction of LAGB is calculated by summarizing the nearest neighbor pixel length with a disorientation angle between  $1.5^\circ$  and  $12^\circ$  and normalized by the total length of the grain boundaries higher than  $1.5^\circ$  but less than  $62.5^\circ$ . The grain boundaries lower than  $1.5^\circ$  are considered to be noise and ignored. The grain size is evaluated by the nearest-neighbor disorientation angle which is higher than  $12^\circ$ . Fig. 1 shows the tri-color inverse pole figure map overlapped with grain boundaries. The above mentioned two types of grain boundaries are drawn in the figure. The bold-black lines indicate the high-angle grain boundaries and the gray lines draw the LAGBs. Fig. 2 shows the statistics of LAGB length fraction of the three samples. It shows that the length fraction of the LAGBs

increases monotonely with reducing the thickness of the specimen. Fig. 3 shows the grain size statistics of three specimens. Some un-recrystallized features can be found in some elongated grains in Fig. 4(a) with intense low-angle grain boundaries. The sample with thickness of 0.3 mm possesses an obvious recrystallized/recovered feature.

#### 3.2. Texture evolution

Though the TiNi alloy texture of the rolling plates and tubes as well as the wires and strips have been studied extensively, however, only a relatively small amount of investigations focused on the textures of ultra-thin rolling sheets integrated with the LAGB and grain size information. The preferred orientations of the ultra-thin hot-rolled sheets differ with the thermal–mechanical heat treatment history and the hot-rolled thick plates. Orientation

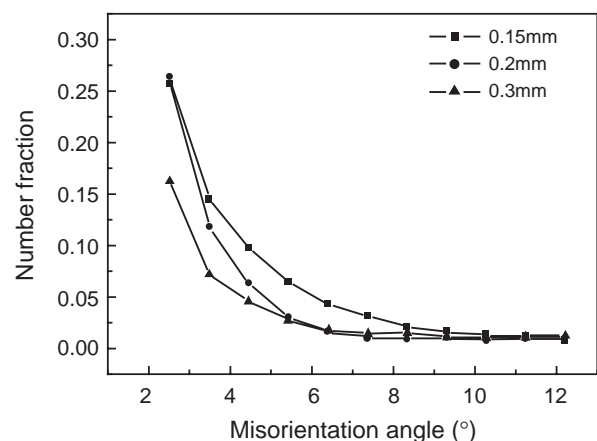


Fig. 2. Low-angle grain boundaries distribution of three specimens with thickness of 0.15, 0.2, and 0.3 mm, respectively.

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