

# Study of residual stress accumulation in TiNi shape memory alloy during fatigue using EBSD technique

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## Abstract

In this study, electron backscatter diffraction (EBSD) technique was applied to study the residual stress accumulation of TiNi shape memory alloy (SMA) during fatigue process, which caused the degradation of the shape memory effect (SME) and super-elasticity (SE). From the analysis of the pattern quality change before and after fatigue test, it showed that, with the stress-induced martensitic transformation (SIMT) during loading process and reverse SIMT during unloading process in each loading cycle, residual stress was accumulated in the parent phase. The results also showed that the residual stress distribution was not uniform inside one grain and between grains. TEM observation showed only dislocation increasing, no persistent slip band (PSB) was found, showing that the non-uniformity of the residual stress distribution was not caused by PSB. © 2005 Elsevier B.V. All rights reserved.

*Keywords:* TiNi; Shape memory alloy; Fatigue; EBSD; Internal stress distribution

## 1. Introduction

TiNi intermetallics are the most attractive shape memory alloys (SMAs) due to their good shape memory effects (SME), super elasticity (SE) and good bio-compatibility, which enable them to be widely used in industry, aerospace, auto-control and medical fields. In many conditions, SMAs are required to work under cyclic stress. Under such circumstance, fatigue failure may occur during service. The fatigue properties of TiNi SMAs are concerned more and more nowadays, many works have been done on the fatigue properties of TiNi SMAs [1–9].

It is known that stress-induced martensitic transformation (SIMT) will take place before slip when an austenitic SMA is deformed. During unloading process, reverse SIMT takes place, SIM will disappear and the strain can be recovered. But this is only under ideal circumstance. The total strain is in fact carried by slip deformation and SIMT together, but the contribution of SIMT is much more remarkable than the other. Shape memory treatment is to constrain the slip deformation and let all the deformation be carried by SIMT. In Ni-rich TiNi SMAs, a

common heat treatment method is quenching+aging for the precipitation of dispersing  $Ti_3Ni_4$  phase which produces certain internal stress field around it so that SIM takes place at lower stress and dislocation movement was constrained at the same time, thus slip is less likely [10]. By this kind of heat treatment, special internal stress field is established and complete SME and SE effects can be achieved. Therefore, the internal stress field is very important to the deformation behavior of TiNi SMAs. Different kinds of internal stress field result in different deformation characteristics. Two-way and all-round SME could even be gained by special thermal–mechanical treatments for establishing special internal stress field [11–13].

During cyclic loading of TiNi SMAs, with the repeated appearance and disappearance of the SIM, residual stress may be accumulated. The residual stress will interact with the previous beneficial internal stress field caused by precipitate  $Ti_3Ni_4$ , causing the continuous change of the internal stress field. The change of internal stress field will influence the SIMT and the deformation mechanism (slip or SIMT) and then the fatigue behaviors. In our previous study [14], it showed that local slip-induced surface coarsening is a main cause of crack initiation of TiNi SMAs. It is necessary to study the disciplinarian of the residual stress distribution in order to clarify the mechanism of the crack initiation of TiNi

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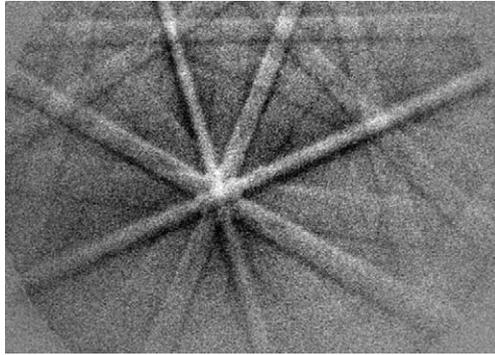


Fig. 1. An EBSD pattern from a B2 TiNi SMA before fatigue. The image quality is very good, showing low internal stress at this point.

SMA during fatigue, but until now, no such work has been reported.

Among all the methods developed to measure the residual stress, X-ray diffraction technique is perhaps the most well developed one and has very high internal stress sensitivity, but its spatial resolution is not good for many applications. TEM technique has the combination of high spatial resolution and stress sensitivity, but the sample preparation is relatively difficult, the stress relaxation during thinning the specimen is a problem as well, and also the analysis field is constrained to a small area so it is impossible to analyse the stress distribution of a large area [15]. Electron backscatter diffraction (EBSD) provides an alternative method for internal stress measurement besides being a powerful tool for the crystal orientation analysis. Since only relatively ideal crystals could generate good Kikuchi pattern quality, residual stress will seriously reduce the image quality of EBSD patterns [15], in this study, the residual stress was measured by checking the pattern quality of the Kikuchi pattern.

## 2. Experimental

The preparation of the experimental materials was described elsewhere [14].

Phase transformation temperatures were measured using differential scanning calorimeter (DSC) method. The  $M_s$  and  $A_s$  are 10 and 17 °C, respectively. At room temperature (25 °C), the material was at austenitic state.

Standard fatigue specimens were applied in the fatigue test. Fatigue tests were carried out using an Instron 8562 fatigue test machine. The waveform chosen for this study is triangle wave. The stress ratio  $R = \sigma_{\min}/\sigma_{\max} = 0$ ,  $\sigma_{\max} = 350$  MPa. After the specimens fractured, the gauge parts were cut using electro-charging machine (ECM) techniques for EBSD analysis. The specimens were heated above  $A_f$  temperature in order to reverse all the SIM back to parent B2 phase and measure the residual stress distribution in parent phase. Specimens for EBSD analysis before and after fatigue test were carefully polished with electro-polishing as the final polishing process. EBSD was carried out as soon as possible to avoid surface contamination. The SEM observation and the EBSD were conducted using a FEI XL30 FEG ESEM with TSL EBSD equipment.

## 3. Results and discussion

### 3.1. EBSD patterns from TiNi SMA before fatigue test

As indicated by the DSC measurement, at room temperature, the samples were in parent phase (B2) state. The experimental materials were solution treated and aged at 400 °C, so the dislocation density was quite low. The EBSD patterns were very nice. Fig. 1 showed one EBSD pattern from TiNi SMA before fatigue test.

### 3.2. EBSD scanning of TiNi SMA before fatigue test

One particular EBSD pattern is generated when the electron beam moves to one position of the surface. The pattern contains orientation information, and each pattern has a particular image quality of the Kikuchi bands. If the electron beam is controlled to scan from point to point continuously in a pre-chosen area, unique grain map, which shows the grain morphology, and the image quality (IQ) map, which approximately shows the internal stress distribution, can be drawn at the same time. The brighter area of an IQ map means lower internal stress in that area, whilst the darker area commonly means bigger internal stress. Fig. 2 shows the unique grain map and the IQ map of a TiNi SMA before fatigue test.

From the IQ map and the unique grain map, it could be determined that the material before fatigue test consisted most of B2 TiNi phase. The grain size is about 40  $\mu\text{m}$ , which is much smaller than that of copper-based SMAs. Smaller grain size is beneficial for relaxing the stress concentration at the grain boundary, thus is beneficial to the fatigue resistance. In the IQ map, it was found that the grain boundary is dark. This is because the grain boundary is the interface of two different grains and the lattice is distorted, so the image quality of the EBSD patterns at these areas is always not good. The grain boundaries in the IQ map are thin dark lines, indicated that there is no extra internal stresses around grain boundaries. It could also be found that the EBSD pattern quality to some extent was controlled by the grain orientation. The gray scale differed somewhat from one grain to another; however, the gray scale of an IQ map was about the same inside one grain. As shown in Fig. 2, it was found that the IQ map was fairly bright, which means the initial internal stress was low before fatigue test.

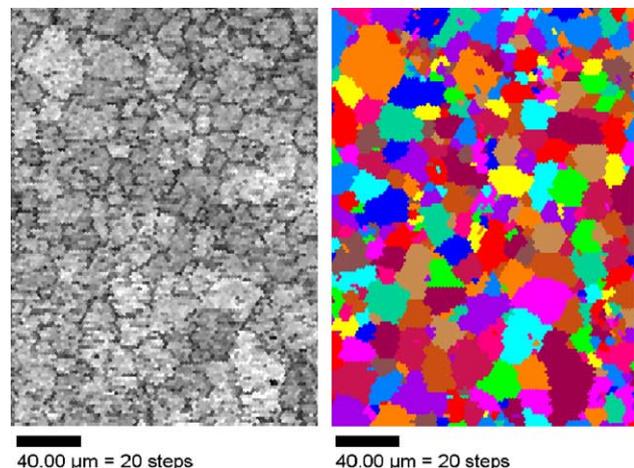


Fig. 2. Image quality (IQ) map (left) and unique grain map (right) of TiNi SMA before fatigue, showing relatively low internal stress before fatigue test from the IQ map and almost full B2 phase from the unique grain map, see the text.

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