



## Full length article

# In situ synchrotron high-energy X-ray diffraction study of microscopic deformation behavior of a hard-soft dual phase composite containing phase transforming matrix



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## ABSTRACT

This study explored a novel intermetallic composite design concept based on the principle of lattice strain matching enabled by the collective atomic load transfer. It investigated the hard-soft microscopic deformation behavior of a Ti<sub>3</sub>Sn/TiNi eutectic hard-soft dual phase composite by means of in situ synchrotron high-energy X-ray diffraction (HE-XRD) during compression. The composite provides a unique micromechanical system with distinctive deformation behaviors and mechanisms from the two components, with the soft TiNi matrix deforming in full compliance via martensite variant reorientation and the hard Ti<sub>3</sub>Sn lamellae deforming predominantly by rigid body rotation, producing a crystallographic texture for the TiNi matrix and a preferred alignment for the Ti<sub>3</sub>Sn lamellae. HE-XRD reveals continued martensite variant reorientation during plastic deformation well beyond the stress plateau of TiNi. The hard and brittle Ti<sub>3</sub>Sn is also found to produce an exceptionally large elastic strain of 1.95% in the composite. This is attributed to the effect of lattice strain matching between the transformation lattice distortion of the TiNi matrix and the elastic strain of Ti<sub>3</sub>Sn lamellae. With such unique micromechanic characteristics, the composite exhibits high strength and large ductility.

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## 1. Introduction

Conventional metallic materials generally have relatively large plasticity due to having massive population of microstructural defects, which facilitate plastic deformation. By the same argument these structural defects render the materials relatively low strength, thus low load-bearing capability, by allowing easy plastic deformation. Some intermetallic compounds and ceramic compounds are able to exhibit higher strengths owing to their stronger interatomic bonding. Low-dimension materials, e.g., nanoparticles, nanowires, and nanoribbons, have been shown to exhibit exceptionally high strength intrinsic to the solids [1–3], generally due to

the lack of structural defects in their crystal. Once again, for the same reason these materials present low ductility due to the absence of effective plastic deformation mechanisms. This has been an intrinsic contradiction and dilemma in materials design for high strength and high ductility.

One possible approach is to combine the attributes of ductile materials and high-strength materials in one composite, and indeed many hard-soft dual phase composites have been designed in the past [4–12]. A common configuration of hard-soft dual phase composites consists of a hard reinforcement embedded in a continuous soft and ductile metallic matrix [5–7]. The hard reinforcement generally serves as the primary load bearer for strength, and the continuous soft and ductile matrix plays the role for load transfer and distribution [5–7].

Whereas the concept seems to be simple and straight forward, the practice is challenging. The essence of such composite design is to achieve synergy between the properties of the two components,

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as opposed to “antergy” (antonym to synergy), and that is where the scientific challenge lies. In conventional hard-soft dual phase metallic matrix composites, the deformation mechanism of the soft phase is primarily dislocation slip [5–7]. Extensive knowledge has been established of the mechanical behavior of conventional metallic matrix composites [13–18]. In the early stage of elastic deformation, load partition between the two components occurs following the rule of mixture. When plastic deformation commences in the matrix, load transfer from the matrix to the reinforcement occurs [16–18], allowing higher load bearing capability of the composite. However, the highly localized lattice distortion of dislocation in conventional metals easily triggers large strain mismatch at the interface, which in turn results in local stress concentration and causes premature local failure of the reinforcement. Consequently, the intrinsic mechanical properties of reinforcement can not be achieved in this conventional composite design strategy [16–18]. This drawback hinders the further improvement of mechanical properties of the composite.

In this work, we applied the recently established nanoscience knowledge at the lattice level to bulk composite design to overcome this challenge. Recently, it is demonstrated that nano reinforcement materials in composites can exhibit ultra-large elastic strains, 5–10 times larger than in bulk form or in conventional metal matrix composites, when embedded in a martensitic phase transforming matrix [19,20]. This unique phenomenon is attributed to the principle of lattice strain matching at the sub-nanometric scale between the transformation lattice distortion of the martensitic matrix and elastic strains of the nanoinclusions. The uniform (and generally lower, <10%) lattice distortion of a martensitic transformation is much easier to coordinate at the interface with the reinforcement than the highly localized lattice strains at a dislocation slip band. This helps to prevent high local stress concentration and assure a relatively uniform stress distribution, thus preventing the premature failure of the rigid component and assuring collective atomic load transfer. This discovery presents a breakthrough to the deadlock of limited elastic strains of nanomaterials in conventional metal matrix composites [19,20] and a new opportunity to design and engineer superior metal matrix composites of exceptional mechanical prowess.

This unique design concept based on lattice strain matching has been demonstrated in a Nb/TiNi nanocomposite system [21]. The composite consists of a polycrystalline TiNi matrix embedded with Nb nanowires of 20–60 nm in diameter all aligned along the axial direction of the composite wire [19,20]. The Nb nanowires in this composite achieved a maximum elastic strain of 6.5%, implying a strength (load bearing) contribution of ~5600 MPa the nanowires within the composite [19,20]. The near perfect axial alignment, the highly dense and uniform distribution and the very large length-to-diameter aspect ratios of the nanowires make the composite a near perfect parallel configuration system in mechanics, i.e. an iso-strain model. Such parallel connection structure facilitates effective lateral load transfer from the matrix to the nanowires, leading to the achievement of ultra-large elastic strains of the nanowires and consequently the high strength of the composite.

Whereas the fore-mentioned Nb/TiNi nanowire composite has proven to be effective in harnessing the exceptional intrinsic properties of the metallic nanowires in composite, the same concept is yet to be validated in other composite structures, such as nanoinclusions in forms other than wires and nanoinclusions that are less orderly distributed within the matrix. Such knowledge is essential for designing and developing metallic composites of exceptional properties based on the lattice strain matching principle. In this work, we investigated the microscopic deformation mechanism of a eutectic Ti<sub>3</sub>Sn/TiNi lamellar composite. The composite provides a unique micromechanical system with distinctive

deformation behaviors and mechanisms from the two components, with the soft TiNi matrix deforming in total compliance via martensite variant reorientation and the hard Ti<sub>3</sub>Sn lamellae deforming predominantly via rigid body rotation as well as elastic deformation.

## 2. Experimental procedure

A 1.5 kg alloy ingot with a nominal composition of Ti<sub>57</sub>Ni<sub>35</sub>Sn<sub>8</sub> (at. %) was prepared by arc melting in a water-cooled copper hearth in an argon atmosphere. Commercial purity Ti (99.99 wt %), Ni (99.99 wt %) and Sn (99.99 wt %) were used as raw materials. The ingot was melted five times in the furnace to obtain chemical composition homogeneity. The composition of the alloy was chosen to conform to a eutectic solidification between TiNi and Ti<sub>3</sub>Sn. The morphology of the composite ingot was characterized using a FEI-200F scanning electron microscope (SEM) operated at 20 kV. The microstructure and the local chemical compositions of the composite ingot were analyzed by means of X-ray energy dispersive spectroscopy (EDS) using a FEI Tecnai G2 F20 transmission electron microscope (TEM). Differential scanning calorimetry (DSC) measurement was conducted using a TA INST2910 differential scanning calorimeter with a heating/cooling rate of 10 °C/min to characterize the transformation behavior of the TiNi matrix. The mechanical properties of the composite were tested in compression using a servo-hydraulic materials testing system (MTS 810) at room temperature at a strain rate of  $5 \times 10^{-4} \text{ s}^{-1}$ . Cylindrical compression samples of  $\phi 5 \times 10 \text{ mm}$  in dimension were prepared according to American Society for Testing and Materials (ASTM) standards.

In situ synchrotron high-energy X-ray diffraction (HE-XRD) measurements were used to study the deformation mechanisms of the composite during compression. A schematic set-up of the measurement is shown in Fig. 1. Experiments were performed on beamline 11-ID-C at the Advanced Photon Source, Argonne National Laboratory, USA. High-energy X-rays with an energy level of 115 keV, wavelength of 0.10798 Å and beam size of  $0.6 \times 0.6 \text{ mm}^2$  were diffracted in transmission geometry towards a Perkin-Elmer large area detector to obtain two-dimensional (2D) HE-XRD diffraction patterns. One-dimensional (1D) HE-XRD diffraction spectrums were obtained by integrating along specified azimuth angles over a range of  $\pm 10^\circ$  in the 2D HE-XRD diffraction patterns. Gaussian fits were employed to determine the positions and areas of the diffraction peaks. The lattice strain for a particular set of crystal planes is calculated as  $|d_{hkl} - d_{hkl}^0|/d_{hkl}^0$ , where  $d_{hkl}^0$  is the “unstressed” lattice spacing (i.e., the peak position at zero applied stress). The errors of the lattice strain measurements and the relative peak intensity measurements were estimated to be less than 0.05% and 0.02, respectively.

## 3. Results

### 3.1. Microstructure of the composite

Fig. 2 shows the structural analysis of the composite created. Fig. 2(a) shows an SEM backscattered electron image of the Ti<sub>3</sub>Sn/TiNi composite. The composite has a full eutectic structure of Ti<sub>3</sub>Sn (bright) and TiNi (gray) phases. The microstructure contained two distinctive morphologies of eutectic cells and inter-cell regions, apparently resulting from the solidification process. The average eutectic cell size is approximately 5 μm. Fig. 2(b) shows the interior of a eutectic cell at a higher magnification. The average lamella spacing is 300 nm. Within the inter-cell regions the average “spacing” is much larger. The volume fraction of the Ti<sub>3</sub>Sn phase is estimated to be about 30% by image analysis. Fig. 2(c) shows a 2D HE-XRD pattern of the composite. It can be seen that the diffraction

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