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# Phase transformation and mechanical properties of as-cast $Ti_{41.5}Zr_{41.5}Ni_{17}$ quasicrystalline composites



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#### ABSTRACT

Phase transformation behavior of the quasicrystalline icosahedral phase (I-phase) and mechanical properties in a Ti<sub>41.5</sub>Zr<sub>41.5</sub>Ni<sub>17</sub> alloy have been investigated using suction-casted bulk samples with various sample sizes ( $\phi = 2$ , 3 and 5 mm). For 2 and 3 mm diameter rod samples, micrometer size dendritic  $\beta$ -(Ti,Zr) phases are precipitated in the matrix and continuously I-phases are developed in the vicinity of the primary  $\beta$ -(Ti,Zr) phases. With further increase of the sample size (i.e. decrease of cooling rate;  $\phi = 5$  mm), the length scale and volume fraction of primary  $\beta$ -(Ti,Zr) dendritic phase are continuously increased and the constitution of matrix phase is changed from I-phase to eutectic lamella composed of bcc  $\beta$ -(Ti,Zr) and hcp C14 Laves phases. The characterization of mechanical properties was carried out by compression and microhardness tests at room temperature. Depending on the selection, size and volume fraction of constituent phases, the values of hardness and compressive fracture strength are varied. The fracture surface morphologies of failed samples exhibit cleavage and quasi-cleavage patterns revealing the typical brittle failure characteristics.

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#### 1. Introduction

Since the quasicrystalline icosahedral (I) phase has been firstly discovered in rapidly solidified Al–Mn alloy by Schechtman et al. [1], a variety of I-phase was found in several alloy systems. The formation of Iphase is very sensitive to solidification process and aging treatment. These I-phases can be divided into two groups based on the thermodynamic stability. One is metastable I-phase [2–11]; the other is thermodynamically stable I-phase [12–15]. Metastable I-phases have been formed in a wide alloy system including Zr- [2–5], Hf- [6,7], Ti- [8–10], Al- [11] and Cu-based alloys [12,13], whereas thermodynamically stable I-phases have been finitely reported in Al–Li–Cu [14], Al–Cu–Fe [15], Ti– Zr–Ni [16] and Zn–Mg–Dy [17] systems.

These stable I-phases have attractive properties such as good corrosion resistance, low friction coefficients, sensitive magnetic properties and hydrogen storage ability. Among them, Kelton et al. found a thermodynamically stable and well-ordered I-phase in the Ti–Zr–Ni ternary alloy [16], which made it possible to widely explore the physical/mechanical properties of the bulk type I-phase. A thermodynamically stable I-phase with the stoichiometry of about Ti<sub>41.5</sub>Zr<sub>41.5</sub>Ni<sub>17</sub> has been obtained by low temperature annealing [16,18]. On the other hand, the stable I-phase has been also developed by rapid solidification process (rapid quenching from the melt). For example, in Ti-(Mn, Fe and Co) binary [19,20] and Ti-Zr-Ni ternary alloys [21,22], ribbon and bulk type I-phases can be fabricated via melt spinning and suction casting methods. Especially, the formability, stability, solidification behavior and kinetics of I-phases have been widely investigated in Ti-Zr-Ni allov [23–25]. Recently, the miner addition of appropriate element can further stabilize the I-phase and improve the I-phase forming ability even in slow cooling rate [26–29]. When cooling rates are properly controlled, the nucleation of Laves-phase can be bypassed, leading to the primary solidification to the I-phase [30]. Therefore, it is of interest to explore the bulk I-phase forming region and understand the influence of the cooling rate on the microstructure and properties of bulk quasicrystalline composite [31]. In the present work, we selected the stable I-phase forming composition (Ti<sub>41.5</sub>Zr<sub>41.5</sub>Ni<sub>17</sub>) and systematically investigated the cooling rate dependent I-phase formability and phase transformation behavior as well as corresponding mechanical properties.

#### 2. Experimental

Alloy ingots with a composition of Ti<sub>41.5</sub>Zr<sub>41.5</sub>Ni<sub>17</sub> were prepared by arc-melting the mixtures of high purity elements (Ti: 99.9995%, Zr: 99.9%, Ni: 99.99%) in a purified argon atmosphere. From the ingots, cy-lindrical bulk samples were prepared by a copper mold suction casting

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Fig. 1. XRD patterns of the as-cast  $Ti_{41.5}Zr_{41.5}Ni_{17}$  alloys with various sample sizes (  $\varphi$  = 2, 3 and 5 mm).

method. The inner cavities in the copper mold have a constant length of approximately 50 mm and their diameters ( $\phi$ ) are 2, 3 and 5 mm. The microstructure of these alloys was examined by a scanning electron



**Fig. 2.** (a) DSC traces obtained during continuous heating of the as-cast  $Ti_{41.5}Zr_{41.5}Ni_{17}$  alloy with a heating rate of 0.33 K/s. Inset shows XRD pattern obtained from the annealed  $Ti_{41.5}Zr_{41.5}Ni_{17}$  alloy up to 880 K. (b) Schematic CCT diagram for the Ti–Zr–Ni alloys. Illustrating i) cooling rate corresponding to the formation of quasicrystalline dual phase composites ( $\varphi = 2-3$  mm) and ii) cooling rate corresponding to the formation of multi-phase composites ( $\varphi = 5$  mm).

microscope (SEM, Jeol JSM-6390) and transmission electron microscope (Tecnai F20). The phase identification was performed by X-ray diffraction (XRD, Rigaku-D/MAX-2500/PC) with Cu K<sub> $\alpha$ </sub> radiation. The phase compositions were determined using a TEM equipped with an X-ray EDX detector. Thin foil samples for TEM analysis were prepared by conventional ion milling (Gatan, Model 600). Phase transformation behaviors of the I-phases were studied by differential scanning calorimetry (Perkin Elmer DSC7). Endothermic event was monitored in the DSC during continuous heating from 800 to 1200 K with a heating rate of 0.33 K/s. To evaluate the mechanical properties, cylindrical samples with a 2:1 aspect ratio were prepared and tested under quasi-static loading. Hardness was measured by the micro-hardness tester with a load of 500 gf. The fracture surface morphologies of failed sample were investigated by SEM.

#### 3. Results

Fig. 1 shows the X-ray diffraction patterns of the as-cast Ti<sub>41.5</sub>Zr<sub>41.5</sub>Ni<sub>17</sub> rod samples with different rod diameters of 2, 3 and 5 mm, respectively. The main sharp diffraction peaks are identified as a mixture of a bcc  $\beta$ -(Ti,Zr) solid solution (*Im*-3*m*), quasicrystalline I-phase and hcp C14 Laves-phase (*P*6<sub>3</sub>/*mmc*). For  $\phi = 2$  and 3 mm rod samples, the diffraction peaks could be analyzed into I-phase with a quasi-lattice constant of  $a_Q = 0.516 \pm 0.0005$  nm and bcc  $\beta$ -(Ti,Zr) solid solution of a = 0.3428  $\pm$  0.0002 nm. With the increase of the sample diameter up to 5 mm, further diffraction peaks were observed, corresponding to the hcp C14 Laves-phase with lattice constants of  $a = 0.5242 \pm 0.0002$  nm and  $c = 0.8581 \pm 0.0002$  nm.



**Fig. 3.** SEM, TEM and SAED patterns of the as-cast  $Ti_{41,5}Zr_{41,5}Ni_{17}$  alloy with 3 mm diameter; (a) cross-sectional SEM BSE image, (b) TEM BF image of the as-cast sample and (c)–(d) SAED patterns obtained from the [122] zone axis of  $\beta$ -(Ti,Zr) and 2-fold symmetry of l-phase, respectively.

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