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Dynamic recrystallization behavior and strengthening-toughening effects in a near-α Ti-xSi alloy processed by hot extrusion

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ABSTRACT

Dynamic recrystallization and strengthening-toughening effects were investigated in the processed near-< alpha > Ti-xSi alloy by hot extrusion. The yielding and ultimate tensile strengths were enhanced by 138.5 MPa and 118.7 MPa in the hot-extruded Ti-Si alloy compared with pure Ti. The double GSS effects (gradient silicon solutes - gradient shearing strain) led to the bimodal microstructure (21.8 μm deformed strips and 5.2 μm recrystallized grains) and grain refinement by dragging the recrystallization nucleation and grains growth during extrusion. The yielding strength enhancement was resulted from multi-factor Si solid solution, grain refinement and oxygen solid solution. Multi-factor Si solid solution was found to be the dominant mechanism, which was a summation of size misfit, modulus mismatch, strain hardening, narrow slip band and texture strengthening. Obvious strain hardening and late necking occurrence would be positive in obtaining the excellent ultimate tensile strength. Ductility was maintained at 22.9% from strain compatibility and cracks suppression. This study may provide a new understanding on the relationship among the mechanical properties, microstructure and processing of Ti-xSi alloy.

1. Introduction

Titanium has been widely used as excellent structural material in many fields due to their promising specific strength, thermal stability, corrosion resistance and biocompatibility [1,2]. However, pure titanium could not meet the increased demand in the mechanical properties with the rapid engineering development [3–5].

Various heavy alloying elements additions, e.g. vanadium, niobium, tantalum and molybdenum, were considered to increase the mechanical behavior by introducing the beta phase [6–9]. While high cost and serious toxicity restricted their applications. Thus, low-cost, environmental-friendly and strengthening alloying element was in highly demand of engineering applications. Silicon, as a low-cost alloying element for titanium alloys, can greatly improve their mechanical/biomedical properties and lower the materials weight, which has been demonstrated by previous researches [10–17]. The biomedical TiNbZrTaSi alloy fabricated by spark plasma sintering was just a very good verification of beneficial effect in the silicon-containing titanium alloys [18]. In fact, silicon is a beta stabilizer (eutectoid-type) and an important strengthening element by substitutional solid solution in the ambient condition. Additive alloying element (silicon) could dissolve into the as-cast titanium in large amounts (~1 wt% in alpha titanium) [11,15,19], showing a strong hardness response and mechanical strengthening on the titanium matrix [20–22]. However, most researcher just focused on the hypereutectic [11,23] and peritectoid [11,15,24] casting Ti-Si alloy. Regretfully, brittle stoichiometric compounds Ti_5Si_3 or Ti_3Si in micrometer-scale should be excluded as poor ductility and moderate strengthening effect. There were some researchers [25,26] trying the casting method to fabricate near-α Ti-Si alloy and they found poor comprehensive properties, which was caused by coarse microstructure and casting defects/impurities. Therefore, available mechanical data of Ti-Si alloy was limited in microhardness, compressive properties or bending properties instead of normal uniaxial tensile properties at the ambient conditions.

It was fortunate that powder metallurgy (PM), the method of fabricating strong and ductile Ti-Si alloy in the present research, was applied with many advantages over the traditional casting methods [27,28]. Recently, in order to restrain excessive oxygen and its deterioration to mechanical properties in the sintering procedure, spark plasma sintering (SPS) was innovatively employed to consolidate pure Ti powders and other alloying element powders [29–34]. The obtained green compacts were then hot extruded (HE) to get full-density materials with excellent mechanical properties [35,36].

However, the fabrication of strong and ductile near-α Ti-Si alloy has never been achieved in the above works. In addition, in the long term of studying Ti alloy fabricated by powder metallurgy and hot extrusion,

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the relevant mechanisms of grain refinement, deformed strips and strengthening-toughening effects were still poorly understood. Therefore, the current work was mainly focused on revealing the mechanisms of grain refinement, and strengthening-toughening effects in extruded near-α Ti-Si alloy.

2. Experimental

2.1. Materials processing/fabrication

Commercially pure Ti powder (irregular shape, mean particle size of 20.6 μm) and pure Si powder were used as the original blending powders. The silicon powder, as the alloying element of addition, presented much finer size about 2–5 μm diameter. Mixing process of these two powders were conducted for 4 h in a rocking mill machine. A predetermined amount of Si addition was mixed in 150 g powders with chemical compositions of Pure Ti, Ti-0.35 wt%Si and Ti-0.70 wt%Si (denoted as Pure Ti, Ti-0.35Si and Ti-0.70Si). The chemical compositions of 0.35Si %mass and 0.70Si %mass were designed according to the Ti-Si binary phase diagram and other references of casting Ti-Si alloy. On the one side, our main purpose of the current work is to study solid solution of Si elements and its influence in the microstructure and mechanical properties of alpha-Ti. On the other side, the intermetallic compound Ti_3Si was a kind of stoichiometric compound (line compound), which was very brittle and harmful to the mechanical properties. Silicon was one of the eutectoid beta stabilizers with a low content of hypoeutectoid reaction (~1% mass). The intermetallic compound Ti_3Si was not expected for the time being, so the chemical composition (far away from the hypoeutectoid point) was designed as 0.35Si %mass and 0.70Si %mass for the preliminary study in the Si solid solution effect. Additionally, the existence of Ti_3Si made it too hard for extrusion even in the high temperature and small extrusion ratio from the view of actual experimental. Of course, the more metallurgical research in the Ti-Si system including the eutectoid reaction with the higher Si content would be developed in the future research. The powder mixture was then sealed in a plastic jar (500 ml in volume) together with ZrO_2 balls (10 mm in diameter). The ball-to-powder ratio was 6:1 and the milling process was carried out continuously in a most simple way. The powder mixture process was conducted by rock milling process (60 Hz, 4 h) in a very low energy. It's very different from the high energy planetary ball milling. The two powders were just mixed together without introducing severe plastic deformation into the powders surface. After milling process the powders surface was not greatly changed. The tested impurity contents included the 0.20% O, 0.03% N, 0.05% Fe, 0.02% C, 0.002% Cr, 0.001% Mn, 0.002%Ni. In order to exclude the impurity of plastic jar and zirconium balls in the milling process, all powders (including pure Ti sample) were taken in the same procedure. So the impurity should not exert big effects on resultant Ti-Si alloy. The powder mixture was then loaded into a cylindrical graphite die and consolidated with a spark plasma sintering system (SPS-1030S, SPS Syntex). The sintering temperature was increased to 1273 K from room temperature in a 20 K/min heating rate and 15 MPa pressure. The compact was sintered at 1273 K for 1.8 ks at a pressure of 30 MPa under vacuum condition of 6 Pa. The green billet possessed a diameter of 42.0 mm and a height of 32.0 mm in geometric. The billet was then surface polished with sand papers to remove the surface impurities and minor oxide layer.

The billet then experienced heat-treatment (Temperature: 1273 K, Duration: 2 h) in the vacuum furnace, which was intended to finish complete solid solution and homogenization at the same time. After heat-treatment the processing flow chart went to the hot extrusion for obtaining the rods. The billet was pre-heated to 1373 K and kept for 300 s under an argon gas atmosphere before hot extrusion. Then, the billet was extruded by a hydraulic press machine (SHP-200–450, Shibayama) with a load capacity of 2000 kN. The extrusion ratio and the ram speed were set at 12.25 and 0.5 mm/s, respectively. The final diameter of the extruded rod was 12.0 mm.

2.2. Measurement and characterization

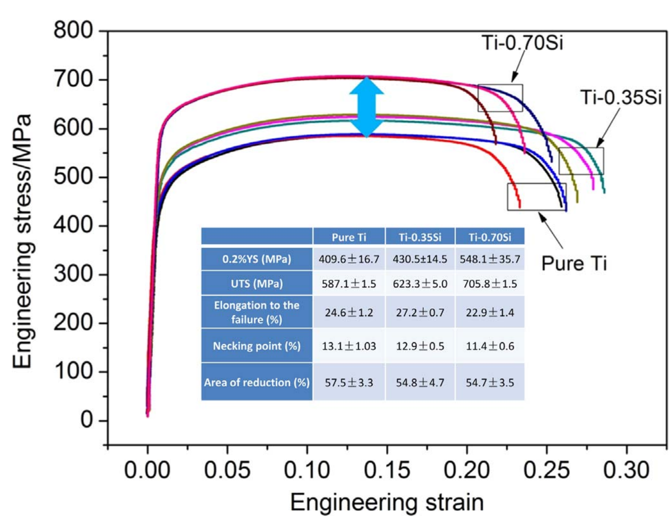
The as-extruded round bar was machined into plate-like tensile test samples with a thickness of 0.50 mm and a gauge length of 10.0 mm along the extrusion direction. Tensile tests (three parallel samples) were conducted on a universal testing machine with a strain rate of 5×10^{-4} s^{-1}. The microstructure and crystallographic orientation were measured by SEM/EBSD system (analysis software: OIM TSL Inc.). The measured surface (longitudinal cross-section) and tensile direction were both paralleling to the extrusion direction. The observation of fine substructure was conducted by FIB/TEM systems. The accurate element analysis was completed with the help of EPMA and TEM-EDS. The post-loading fractographs observation was conducted by SEM (FESEM, JSM-6500F, JEOL). The oxygen and nitrogen contents of the extruded Ti-xSi materials were measured with a nitrogen–oxygen instrument (TC-300, LECO).

3. Results

3.1. Mechanical properties

The room-temperature tensile tests were carried out to measure the mechanical properties of the as-extruded Ti-xSi alloys. The obtained engineering stress–strain curves and concluded mechanical properties were demonstrated by Fig. 1. With increasing the silicon addition, the strength was greatly enhanced and the ductility was maintained at a relatively high standard concurrently. Obviously, the high strength-toughness titanium alloy is successfully obtained by this method, which is most valuable in the engineering application due to its easy processing routes and excellent comprehensive properties.

It was more interesting to dig deep the detailed tensile curves. These mechanical data contained strength (yielding strength and tensile strength), ductility (elongation to the failure and fracture area of reduction) and onset strain of necking (i.e. strain hardening exponent). Firstly, with the addition of 0.70% Si the double strength (YS and UTS) of Ti material got great enhancement (YS: 33.8%, 138.5 MPa; UTS: 20.2%, 118.7 MPa). At the same time, it's exciting to find that ductility was just slightly decreased by 1.7%. Fracture area of reduction included necking signal in the plasticity and absorption of fracture energy, which was better and supplementary information to show its high ductility of Ti-Si samples. From the projected area of S-S curves, toughness of Ti-0.70Si was obviously greater than that of pure



	Pure Ti	Ti-0.35Si	Ti-0.70Si
0.2%YS (MPa)	409.6±16.7	430.5±14.5	548.1±35.7
UTS (MPa)	587.1±1.5	623.3±5.0	705.8±1.5
Elongation to the failure (%)	24.6±1.2	27.2±0.7	22.9±1.4
Necking point (%)	13.1±1.03	12.9±0.5	11.4±0.6
Area of reduction (%)	57.5±3.3	54.8±4.7	54.7±3.5

Fig. 1. Engineering strain-stress curves and conclusive tensile properties of extruded Ti-xSi samples.