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ARTICLE

Effects of Composite Processing at Room Temperature and Heat Treatment on Microstructure and Mechanical Properties of Ti-53Nb Alloy

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Abstract: The bars of Ti-53Nb alloy were prepared by combined deformation method of room temperature equal channel bending channel deformation (ECAP)+cold rolling+cold rotary swaging. The evolution of microstructure and β-crystal growth were studied by metallographic microscope (MM), scanning electron microscope (SEM) and uniaxial tensile test after preparing process. The effects of work hardening and fine grain strengthening were analyzed with dislocation strengthening theory and Hall-Petch theory. The results show that the tensile strength increases from 380 MPa to 553 MPa before and after deformation and heat treatment, increased by 45.53%, and the elongation reaches 16%. The growing speed of β-crystal is accelerated with increasing the solid solution temperature, and the grain size dependence of strengthening follows the Hall-Petch equation. The microstructure exhibits uniform equiaxed grains after annealing at 700 °C for 60 min, which can match the requirement of strong plastic characteristics in applications.

Key words: ECAP; rotary swaging; Ti-53Nb; solution treatment; microstructure and performance

Ti-Nb alloy has received much more attention due to its potential application in biomechanical matching material[1-3]. The components of Ti and Nb have good biocompatibility; Nb element, as a β-stabilizing element, can reduce the elastic modulus of the alloy. Ti-53Nb alloy is a binary stable β-titanium alloy with a body-centered cubic (bcc) structure, and has significantly more slipping system than α-type titanium alloy with a close-packed hexagonal structure, thereby providing more possibilities for slipping deformation. However, the potential application is still restricted due to its lower tensile strength compared to that of the duplex titanium at room temperature.

Equal channel bending channel deformation (ECAP) is a strong plastic deformation technology, which can refine the grain size and improve the strength and plasticity of the material[4,5]. ECAP technology is currently applied to prepare pure titanium, zirconium, zirconium alloys and aluminum alloys. The technology of cold rolling and cold rotary swaging supplies the 3D (dimensions) compressing stress machining method. The composite application of two techniques can not only refine the grains, but also increase the straighten, which has been confirmed by Ref.[6]. The equal channel angular extrusion (ECAP)+cold rolling (CR)+cold rotary swaging (CRS) composite processing can significantly improve the strength of pure titanium with uniform distribution[7]. The single pass of industrial pure zirconium is successfully prepared by a conventional equal-diameter curved channel deformation (ECAP) mold (channel angle 90°, internal fillet 20°). The grains are significantly broken and the yield strength and tensile strength are 397 and 536 MPa, increased by ~43% and ~53%, respectively[8]. The ultrafine titanium from industrial pure titanium is obtained

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through one pass ECAP deformation and cold rolling composite deformation at 105° mold. The average grain size of ultrafine titanium is about 130 nm with the tensile strength as high as 813 MPa. In the annealing temperature regime less than 400 °C, no obvious change occurs in the microstructure, but the strength and hardness decrease slowly, and the increasing tendency of elongation tends to stop[9]. For the stable binary β titanium alloy, the deformation strengthening technology is one of the very effective methods to improve the strength of the material.

In order to improve the strength of Ti-53Nb alloy, the preparing process was designed as ECAP+cold rolling+cold rotary swaging at room temperature to study the microstructure evolution and mechanical properties under the room temperature deformation condition. Based on the deformation at room temperature, the effect of heat treatment on the static recrystallization behavior was studied, and the effect of solid solution system on the growth behavior of β grains and the recrystallization kinetics were analyzed.

1 Experiment

The rod of Ti-53Nb alloy with compositions of Nb-52.5, C-0.009, H-0.0026, O-0.034 and balance Ti, which was developed by Northwest Institute for Nonferrous Metal Research, was used as the raw material.

Firstly, one cylindrical sample for the initial forging rod was polished with a diameter of 24 mm for matching the hold size of ECAP (diameter of 25 mm, channel angle of Φ=135°, and rounded angle of ψ=20°). Two passes of room temperature extrusion deformation were performed at a speed of 3.5 mm/s in the mold. The preferred extrusion path was along c-axis, that is, the initial pass was rotated 180° around the longitudinal axis of the sample. Secondly, the lubricant was a mixture of ultrafine graphite powder in water. The extruded sample was repeatedly deformed on a cold rolling mill until the rectangular sample of 15 mm×15 mm was rolled with a cumulative reduction amount of 40%. Finally, the forging process was performed on a DH7-4III precision forging machine to obtain a rod-shaped sample with a diameter of Φ8 mm and a smooth surface without cracks. The sample appearances are shown in Fig.1.


As-received ECAP CR CRS

Fig.1 Appearances of Ti-53Nb alloy after ECAP+CR+CRS processing at room temperature

2 Results and Discussion

2.1 Evolution of deformed microstructure at room temperature

The morphology of Ti-53Nb alloy before deformation is shown in Fig.2a. The microstructure is composed of equiaxed crystals without deformation or shear band. Fig.2b shows the morphology of Ti-53Nb alloy after two passes of ECAP extrusion. It can be seen that the internal slip zone of the original grains does not increase, and some grains are elongated and deformed, which can increase opportunity of the combined action of slip and grain boundary slide. The microstructure shows that some grains are broken and associated with present a large number of fibrous shear bands during dynamic deformation process. With the increase of the slip zone, the dislocation density tends to increase, and causes the blurry at grain boundaries, as shown in Fig.2c. The cold rotary swaging deformation induces stretching, thinning and twisting phenomena for the grains, and fibrous bands form at the grain boundaries, as shown in Fig.2d. This morphology is described as “Van Gogh’s Sky (VGS)”[10], which often occurs during the process of room-temperature deformation for bcc type metals with a large deformation. This morphology is suggested as a typical vortex-like microstructure[11] originated from (110) plane strain, where β fiber texture and grain boundary coexist.

2.2 Mechanical properties at room temperature

Fig.3a shows the strain dependence of tensile stress for various deformed alloys at room temperature, and the tensile properties and its relative change is shown in Fig.3b. It can be seen that in the initial state, the alloy has a low tensile strength of about 380 MPa with an elongation of 27.5%. After the deformation, the tensile strength and yield strength of the alloy continue to increase. The tensile strength increases from 380 MPa to 477 MPa with an increase of 25.5%, and the elongation is reduced from 27% to 19% after processing by ECAP at room temperature in the initial state. The room temperature deformation causes the increase of dislocation density, the interactions between dislocation cut order and dislocation entanglement increases, and the resistance of dislocation movement also increases; the dislocation strengthening effect is significant, and the alloy exhibits a significant work hardening phenomenon. With the increase of cumulative deformation, the tensile strength also enhances. The cold rotary swaging deformation induces the increase of tensile strength (553 MPa) for the alloy, increased by 45.53% compared to that of the original one, implying that the dislocation strengthening effect is obvious due to the composite deformation at room temperature, and the elongation reaches 16%; the coarse original grains are broken under the action of shearing and torsional forces, forming a large number of crystal defects, hindering the movement of dislocations, and achieving the purpose of dislocation strengthening[12].

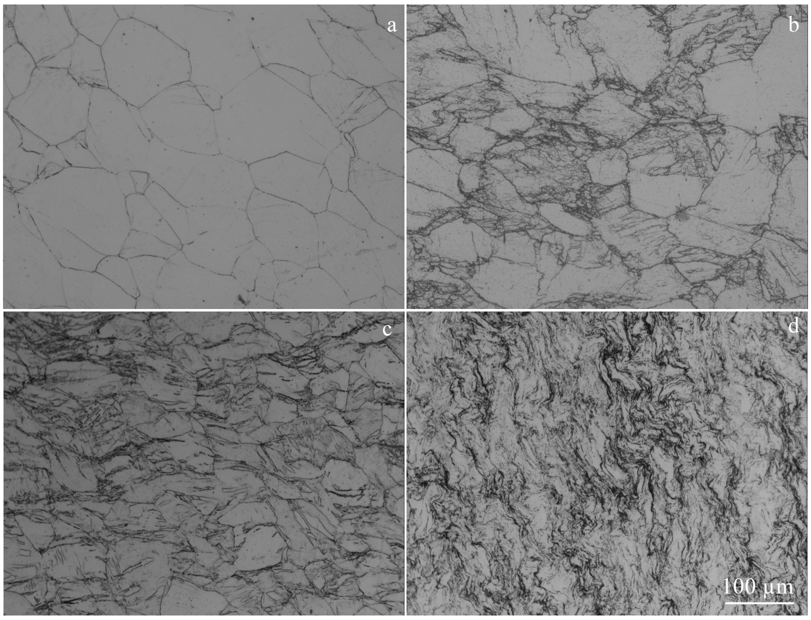

Fig.2 Optical microstructures of Ti-53Nb alloy after different deformation methods: (a) as-received, (b) two pass of ECAP at room temperature, (c) cold-rolling, and (d) cold rotary swaging

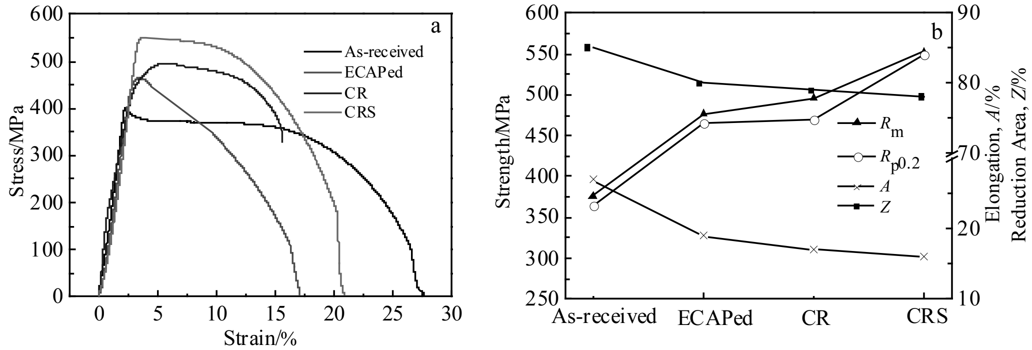

Fig.3 Stress-strain curves (a) and mechanical properties (b) of the Ti-53Nb alloy after deformation

The tensile fracture morphologies of Ti-53Nb alloy before and after deformation is shown in Fig.4. It shows that the deformation causes the reduction of area under low magnification, but there is no effect on the severe necking and good plasticity is observed. The shear lip is at an angle of 45° to the direction of the tensile stress, indicating that dislocation slip occurs during the stretching process. As shown in Fig.4b, the thick and deep dimples are distributed between the tearing ridges, showing a way of trans-granular fracture. The formation of this microstructure is due to no pinning effect of α phase at the grain boundaries in the Ti-53Nb alloy and no obstacle to the external growth of the grains. The size of the dimples mainly depends on the grain size. During the process of deformation, the grains are obviously elongated and cause the smooth depression for the dimples. The size of the dimples is reduced, and it might be related to the refinement of the grains caused by severe deformation. In addition, the stress concentration forms at the intersection of the sliding surfaces due to the room temperature deformation.

2.3 Microstructure after solid solution

Fig.5 shows the microstructure of the deformed alloys after recrystallization and solid solution process at 500, 600, 700, 800 and 900 °C for 60 min. It can be seen that the average grain size enlarges with increasing the temperature, and the equiaxed crystals finally form after solid solution heat treatment at 900 °C for 60 min. The phase dissolves at 500 °C, showing that grain boundary is melted and it is still streamlined. However, the recrystallization nucleation cannot occur at 500 °C. At this stage, the recovery of the alloy mainly occurs, which induces the decrease of dislocation slipping, climbing and system strain energy. The fine grains crystalize at the boundary of the deformation streamline at 600 °C, but the distribution is nonuniform. With continuously increasing the temperature to 700 °C, the static recrystalliza-

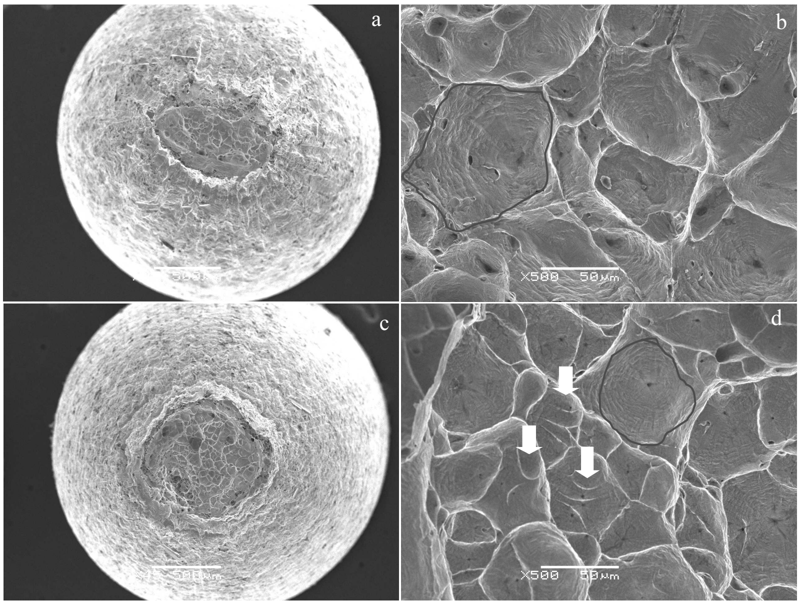

Fig.4 Tensile fracture morphologies of the Ti-53Nb alloy before (a, b) and after (c, d) deformation

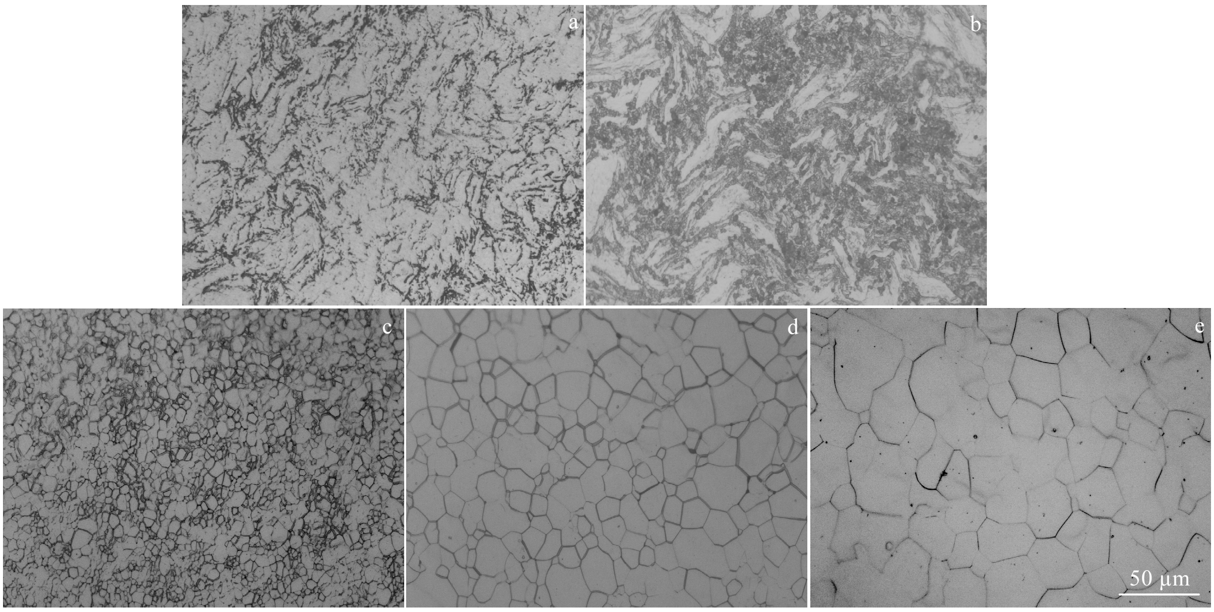

Fig.5 Effect of solution temperature on microstructures for Ti-53Nb alloy: (a) 500 °C, (b) 600 °C, (c) 700 °C, (d) 800 °C, and (e) 900 °C

tion basically forms, and the uniformed fine equiaxed grains appear. With increasing the temperature to 800 °C, the grain size increases significantly, but its distribution is not uniform and there are even abnormally grown grains.

In the same strain case, the accumulated distortion energy of the alloy is equivalent. If the static recrystallization temperature is reached, the nucleation is completed quickly in the large distortion region, and the recrystallization nucleation grow is driven by atomic diffusion. The effect of solid solution temperature on grain growth mainly depends upon the atomic migration and diffusion process at the surface and interfaces of grain boundary in the alloy, and the solute atoms crossing the alloy interface are considered as a thermally activated process. The average mobility at the grain boundaries is proportional to index ($-Q/RT$). The higher the temperature, the faster the grain growth rate, and the higher the corresponding grain growth index[13].

As the solution temperature increases from 800 °C to 900 °C, the average grain size in the alloy increases from 17 μm to 35 μm, and the growth trend is obvious. It can be seen that the speed of grain growth continuously increases with the increase of temperature in the same holding time.

2.4 Effect of solution temperature on mechanical properties

The mechanical properties of Ti-53Nb alloy processed at various solid solution temperatures (500, 600, 700, 800, and 900 °C) for 60 min are shown in Fig.6. It can be seen that the ultimate tensile strength (R_m) of alloy, which is processed at solid solution temperature of 500 °C, nearly decreases by 30 MPa compared to that of one processed by cold rotary swaging, but the elongation increases. This phenomenon is due to the decrease of dislocation density. The effect of dislocations on preventing further deformation is weakened during tensile deformation[14]. The grains are refined by accelerating the grain boundary movement at 700 °C, which causes weak interaction between dislocations and improves the plasticity of the alloy.

In high temperature region, the growth of β phase is fast, which can improve the tensile strength and elongation of the alloy.

2.5 Recrystallization kinetics

The growth of grains for β phase depends on the composite effects of kinetics and thermodynamics. The kinetics of growth should meet the reduction of the total interfacial energies, while the thermodynamics is built based on the solution temperature and holding period. The higher the temperature, the faster the diffusion rate of atom crossing the interface, which is more favorable for the growth of grains; the longer the holding time, the more the atoms diffuse, and the larger the driving force of the grain growth [15].

During the heat treatment process for the β grain recrystallization at various solid solution temperatures within the same period, The grain size D follows the Arrhenius equation, as listed in Eq.(1)[16].

$$\bar{D} = A\exp(-Q_g / RT) \quad (1)$$

where Q_g is the activation energy for grain growth (kJ/mol); T is the absolute heating temperature (K); R is the gas constant;

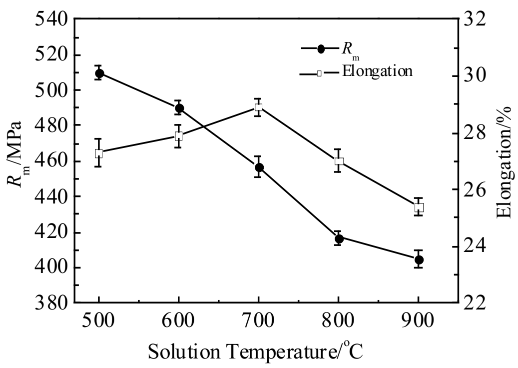

Fig.6 Relation between solution temperature and mechanical properties of the Ti-53Nb alloy

A is the proportionality constant. The linear reversal T dependence of logarithmic of size is obtained by logarithmic transformation for Eq.(1), as listed in Eq.(2):

$$\ln \bar{D} = \ln A - Q_g / RT \quad (2)$$

The observed $1/T$ dependence of ln$D$ follows Eq.(2) well, as shown in Fig.7. The fitted parameters correspond to 6946.07 for the slope Q_g/R and 9.2 for the intercept ln$A$. The fitted active energy Q_g is about 57.7 kJ/mol for Ti-53Nb alloy grain growth, which is higher than that of pure titanium and other titanium alloys due to the high Nb content [17].

In summary, the kinetics of grain sizes follows the fitting equation listed in Eq.(3) for Ti-53Nb alloy prepared by the solid solution heat treatment in temperature range of 600~900 °C for 60 min.

$$\bar{D} = 9897.1\exp(-5.77\times10^3 / 8.314T) \quad (3)$$

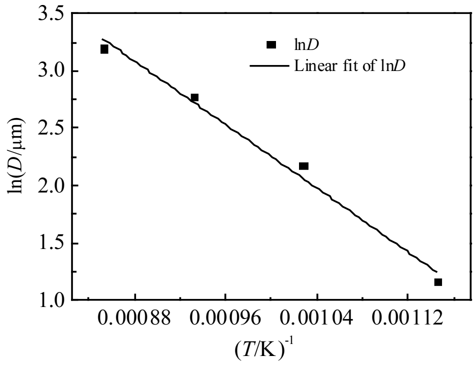

Fig.7 Curve of ln$D$-1/T for the alloy at various solution heat treatment temperatures

3 Conclusions

1) Ti-53Nb alloy can be significantly strengthened by room temperature deformation by compositing technologies of ECAP+cold rolling+cold rotary swaging. The strength can reach 553 MPa with an increase ratio of 45.53% and the elongation can reach 16%. The microstructure shows a typical spiral fiber structure with dislocations.

2) The equiaxed grains with strong plastic properties is obtained by solid solution heat treatment at 700 °C for 60 min.

3) The kinetic characteristics of grain growth follows the formula of $\bar{D} = 9897.1\exp(-5.77\times10^3 / 8.314T)$. The fitted activation energy is 57.7 kJ mol for Ti-53Nb alloy at the heat treatment temperatures of 600~900 °C for 60 min.

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室温复合加工及热处理对 Ti-53Nb 组织及性能的影响

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摘　要：采用室温 ECAP+冷轧+冷旋锻复合变形方法制备了 Ti-53Nb 合金棒材，通过金相显微镜、扫描电子显微镜、单向拉伸试验等研究了合金室温及热处理后组织演变及 β 晶粒的长大行为，并分析了加工硬化和细晶强化效应。结果表明：室温抗拉强度由变形前的 380 MPa，提升到了变形后的 553 MPa，提高了 45.53%，延伸率也在 16%以上。随固溶温度升高，β 晶粒长大速率加快；晶粒尺寸对合金的强化作用满足 Hall-Petch 关系式。在 700 ℃，保温 60 min 的条件下，组织均匀呈细小等轴状，可以获得良好的强塑性匹配。

关键词：ECAP；旋锻；Ti-53Nb；固溶处理；组织与性能

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