TECHNICAL ARTICLE

Effect of Ti on the Microstructure and Mechanical Properties of AICrFeNiTi_x Eutectic High-Entropy Alloys

Songyuan Li, Feida Chen _®, Xiaobin Tang, Guojia Ge, Zhangjie Sun, Zhenlong Geng, Minyu Fan, and Ping Huang

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High-entropy alloys (HEAs) have become important candidates for structural materials of nuclear reactors because of their excellent mechanical properties and irradiation resistance. Recently, eutectic HEAs (EHEAs) have exhibited improved properties by double-phase strengthening. In this study, the effect of Ti on the microstructure and mechanical properties of the EHEAs AlCrFeNi was investigated. Results show that the eutectic microstructure consisted of a disordered body-center cubic (A2) phase and an ordered body-center cubic (B2) phase. Smaller nanoparticles were formed with Ti addition. Ti significantly influenced the microstructure of AlCrFeNi, thus changing the mechanical properties of AlCrFeNi. The compressive strength of AlCrFeNi increased, and the toughness of AlCrFeNi decreased with the increase in Ti content. The application of $AICrFeNiTi_{0.2}$ alloy is promising because of its high mechanical properties and superior specific strength. In addition, the $AICrFeNiTi_x$ alloys showed great softening resistance at a high temperature (500 °C).

1. Introduction

Nuclear energy is critical for the future energy system due to its low carbon emission (Ref [1\)](#page-8-0). Compared with the existing reactors, the Gen-IV reactors have high energy efficiency, low pollution, and a promising future (Ref [2\)](#page-8-0). The structural materials for Gen-IV reactors endure high temperatures, high irradiation doses, and severe corrosion, resulting in significant volume swelling, segregation, and embrittlement of structural materials (Ref [3](#page-8-0)). Many investigations have devised new materials, such as high-entropy alloys (HEAs), oxide dispersion steel (ODS), and FeCrAl alloy (Ref [4-7\)](#page-8-0), to meet the requirements of the Gen-IV reactors.

HEAs are multi-principal element alloys composed of five or more elements in an equal atomic ratio or nearly equal atomic ratio (Ref [8\)](#page-8-0). Many studies have proven that HEAs have high strength, exceptional hardness at elevated temperatures, and excellent corrosion resistance (Ref [9-16](#page-8-0)). Furthermore, previous studies have emphasized that the HEAs have excellent irradiation resistance compared with conventional alloys owing

Songyuan Li, Feida Chen, Guojia Ge, Zhangjie Sun, and Zhenlong Geng, Department of Nuclear Science and Technology, Nanjing University of Aeronautics and Astronautics, Nanjing 211106, China; Xiaobin Tang, Department of Nuclear Science and Technology, Nanjing University of Aeronautics and Astronautics, Nanjing 211106, China; and Key Laboratory of Nuclear Technology Application and Radiation Protection in Astronautics, Ministry of Industry and Information Technology, Nanjing 211106, China; and Minyu Fan and Ping Huang, Suzhou Nuclear Power Research Institute, Suzhou 215004, China. Contact e-mail: fdchen@nuaa.edu.cn.

to their unique lattice distortion effect. Therefore, HEAs are considered the important candidates for structural materials of nuclear reactors (Ref [17-19\)](#page-8-0). Eutectic HEAs (EHEAs), as a special kind of HEAs, have low-energy phase boundary, high rupture strength, stable defect structure, and good hightemperature creep resistance (Ref [20-22\)](#page-8-0). These features ensure the application of EHEAs in high-temperature environments (Ref [23](#page-8-0), [24](#page-8-0)).

As one of the first known EHEA systems, Al-Co-Cr-Fe-Ni has outstanding strength and ductility (Ref [25,](#page-8-0) [26](#page-8-0)). Lu et al. (Ref [23\)](#page-8-0) proposed AlCoCrFeNi_{2.1}, composed of alternating soft FCC and hard BCC phases, showing an unprecedented combination of high ductility and fracture strength. Lim et al. (Ref [27](#page-8-0)) studied the mechanical properties of AlCoCrFeNi at high temperatures. The alloy showed superior specific strength to TiAl and titanium alloys in the temperature range from 773 to 873 K. Composition optimization design is an effective way to enhance the microstructure and mechanical properties of the alloy. A large number of studies have focused on the addition of microalloying elements. Previous results (Ref [28\)](#page-8-0) show that Ti has the following merits, including low density, high melting point, and large atomic radius, which can improve the mechanical properties of alloys. Chen et al. found $Ti_{0.15}AlCoCrFeNi_{2.1}$ alloys showed the most excellent combination of high ultimate tensile strength (1253 MPa) and ductility (12.9%) (Ref [28\)](#page-8-0). Similarly, Munitz et al. added 11.1% Ti to AlCrFeNi to create AlCrFeNiTi_{0.5}, which increase the alloy strength from 1560 to 1660 MPa (Ref [29\)](#page-8-0). Most recently, Elyorjon Jumaev et al. found the yield strength was improved from 1389 to 1703MPa and high-temperature hardness was also enhanced after the addition of 5 at.% of Ti to AlCrFeNi (Ref [30\)](#page-8-0). These researches suggest that the HEAs through Ti addition exhibit excellent performance. However, a systematic investigation of microstructure and mechanical properties of alloys caused by Ti addition is deficient. In addition, the element Co in the system has high neutron transmutation-induced radioactivity, which makes it unfavorable for nuclear applications. Thus, using Ti instead of Co to obtain high-performance

Al-Cr-Fe-Ni-Ti alloy and exploring the ideal content of Ti element are the cores of this study.

On the basis of the abovementioned discussion, AlCrFeNi- Ti_x (x=0, 0.2, 0.5, 1) alloys with different Ti contents were prepared. The effects of Ti content on the phase and microstructure of the material were systematically studied. The variations in compression strength and hardness under different Ti contents were also investigated.

2. Experimental

AlCrFeNiTi_x ($x=0$, 0.2, 0.5, 1) alloys were prepared by cold crucible levitation melting under argon atmosphere, and they were denoted as "Ti0," "Ti0.2," "Ti0.5," and "Ti1" hereinafter. Before melting, the furnace was kept below the pressure of 7×10^{-1} ³ Pa, and protective argon through the furnace was maintained under the constant pressure of 0.05 MPa. The purity of raw metals is higher than 99.95%. The ingots were slipped and remelted at least four times to ensure chemical homogeneity and finally became ingots with a mass of 200 g. The ingot was cut into $10 \times$ 10×1 mm³ thin slices by wire-electrode cutting, abraded on SiC paper up to 2000 grit, and polished for subsequent characterization. Parts of the samples were subjected to heat treatment under a pressure of 1×10^{-4} Pa to investigate the softening resistance at a high temperature. To meet the requirement of sodium fast reactor (SFR), 500 °C was chosen as annealing temperature (Ref 31). The temperature increased from room temperature to 500 $^{\circ}$ C at a rate of 30 °C/min and reduced to 100 °C at a rate of 3 °C/min after being homogenized at 500 $^{\circ}$ C for 3.7 h.

Rigaku Ultima-IV x-ray diffraction (XRD) with Cu-Ka radiation was used to characterize the crystal structure and phase composition of the samples. The 2θ scanned from 20° to 100° with a speed of $5^{\circ}/\text{min}$. The microstructure was investigated by scanning electron microscope (SEM, TESCAN LYRA3) equipped with energy-dispersive spectrometry (EDS). The thermal behavior was analyzed by differential scanning calorimeter (DSC, NETZSCH STA 449F3) from room temperature to 1500 \degree C with a rate of 20 \degree C/min under Ar atmosphere. The microstructure and phase were characterized by transmission electron microscope (TEM, FEI Talos F200X). The TEM sample was prepared by mechanical polishing to about 60 μ m thickness, followed by dual jet polishing in ethanol solution containing 9% HClO₄.

Cylinder specimens with 5 mm in diameter and 10 mm in height were prepared for compression tests. The tests were conducted at room temperature using WDW-100 with a strain rate of 1×10^{-3} s⁻¹. The Vickers hardness was tested under a load of 9.8 N for 10 s. In addition, the nano-indentation hardness of the samples was measured on Nano Indenter G200. The continuous stiffness measurement with a Berkovich-type indenter was adopted in the tests. The indentation depth of 1.7 μ m and the interval of 34 μ m were set for the sample. Each sample was tested five times to reduce errors.

3. Results and Discussion

3.1 Phases and Microstructure

Figure 1 shows the XRD patterns of as-cast $AICrFeNiTi_x$ alloys. The phase without Ti addition is composed of disor-

Fig. 1 XRD pattern of as-cast AlCrFeNiTi_x alloys

dered BCC (A2) and ordered BCC (B2). The overlapping diffraction peaks $((110), (211), (220))$ of the BCC phase indicate that the lattice constants of A2 and B2 are similar. When the Ti content is less than 11 at.%, no diffraction peak of other phases in addition to the A2 and B2 is found. The diffraction peaks of A2 and B2 shift to the left slightly with Ti addition, indicating that the lattice constant increases after Ti addition. The lattice constants of the B2 phase are 2.909, 2.910, 2.952, and 2.930 Å for Ti0, Ti0.2, Ti0.5, and Ti1, respectively. The lattice constants of the A2 phase are 2.903, 2.899, 2.919, and 2.898 Å for the alloys. Ti1 also forms some intermetallic compounds, such as $L2₁$ phases. This finding is consistent with the results of previous studies (Ref [21\)](#page-8-0).

The microstructures of AlCrFeNiTi_x characterized by SEM are presented in Fig. [2](#page-2-0). The four alloys have eutectic microstructures, which differ with different Ti additions. As presented in Fig. [2](#page-2-0)(a), the net-like eutectic microstructure occurs in Ti0. Each grain is composed of two regions: the core and the periphery. Two phases with different contrasts are observed in both regions. The high-magnification images in Fig. [2\(](#page-2-0)b) show the periodic plate-like microstructure in the core, which is a typical feature of spinodal decomposition (Ref [32,](#page-8-0) [33](#page-8-0)). The morphology difference between the core region and the periphery is the least in Ti0 among all four alloys. The finer phase structure and more dense phase boundaries are formed in Ti0 than in other alloys. The ratio of the two phases is close to 1:1. As shown in Fig. [2\(](#page-2-0)c), a typical net-like eutectic structure appears in Ti0.2, which gradually becomes larger from the central part to the periphery. In addition, the structure is composed of radiating lamellar phases, where nanometer-sized precipitates are distributed uniformly. The morphology of Ti0.5 (Fig. [2e](#page-2-0)) is similar to that of Ti0.2. The area of the phase with dark contrast in the periphery becomes larger, while the Ti element is added. Ti1 contains lamellar and cellular eutectic microstructure, and the lamellar thickness is less than 100 nm. In addition, the cellular microstructure is surrounded by fine lamellar microstructure. Figure [2\(](#page-2-0)h) shows the enlarged morphology of the interface between the two kinds of eutectic microstructures. Nanoparticles marked with a yellow circle distribute uniformly in the center, and the size of nanoparticles was smaller than that of the particles in Ti0.5. Considering that

Fig. 2 SEM images of as-cast AlCrFeNiTi_x alloys: (a) Ti0, (c) Ti0.2, (e) Ti0.5, (g) Ti1; (b), (d), (f), and (h) are the enlarged images of (a), (c), (e), and (g), respectively

Table 1 Chemical composition of the marked regions in Fig. [2](#page-2-0) analyzed by EDS

Element	Al, at. $%$	Cr, at. %	Fe, at $%$	Ni, at. $%$	Ti, at. $%$
Point 1	31.17 ± 1.6	18.83 ± 0.6	19.39 ± 0.8	30.62 ± 1.2	\cdots
Point 2	12.12 ± 0.8	35.62 ± 1.2	36.34 ± 1.3	15.91 ± 0.8	\cdots
Point 3	13.31 ± 0.7	36.33 ± 1.2	34.55 ± 1.3	13.19 ± 0.7	2.63 ± 0.2
Point 4	28.25 ± 2.1	22.19 ± 1.3	17.54 ± 1.2	23.87 ± 1.7	8.15 ± 0.7
Point 5	22.29 ± 1.3	16.62 ± 0.7	19.68 ± 0.8	27.07 ± 1.2	14.34 ± 0.5
Point 6	10.05 ± 0.6	47.56 ± 1.6	32.31 ± 1.2	5.58 ± 0.4	4.50 ± 0.2
Point 7	14.48 ± 0.7	43.36 ± 1.1	31.20 ± 1.0	5.99 ± 0.3	4.97 ± 0.2
Point 8	24.36 ± 1.2	10.28 ± 0.4	17.21 ± 0.6	30.53 ± 1.1	17.61 ± 0.6
Point 9	22.71 ± 1.4	11.47 ± 0.6	17.53 ± 0.9	26.29 ± 1.3	22.00 ± 0.9
Point10	13.57 ± 0.7	52.08 ± 1.6	19.68 ± 0.8	4.81 ± 0.3	9.86 ± 0.4

Fig. 3 TEM images and SAED patterns along [01 1] zone axis of as-cast AlCrFeNi. (a) Bright-field image. (b) and (c) are the SAED patterns of bright and dark regions shown in (a), respectively. (d) shows EDS elemental mapping

Fig. 4 Differential scanning calorimeter (DSC) curves for AlCrFeNiTix

Fig. 5 Compression stress–strain curves of AICrFeNiTi_x at room temperature

the cooling rate can influence the microstructure during the solidification, we speculate Ti can influence the cooling rate. The microstructure morphology transforms from lamellae to petals with the addition of Ti, which is consistent with a previous study (Ref [28](#page-8-0)).

The chemical compositions of the marked regions in Fig. [2](#page-2-0) are summarized in Table [1](#page-3-0). In accordance with the chemical composition in Table [1](#page-3-0), region 1 contains a large amount of Al and Ni, when region 2 contains a large amount of Fe and Cr. Based on previous studies, Ni-Al tends to form the B2 phase, while Fe-Cr tends to form the A2 phase (Ref [33](#page-8-0), [34](#page-8-0)). Therefore, region 1 should be corresponding to B2 phase and region 2 should be corresponding to A2 phase. Furthermore, Ti segregates in the regions abundant in Ni, Al because of the negative enthalpy of mixing between Ti and Ni (Ref [35](#page-8-0)). EDS results confirm that regions 6, 7 are enriched in Cr and Fe, and regions 5, 8 are enriched in Al, Ni, and Ti. Therefore, regions 6, 7 are A2 phases, and regions 5, 8 are B2 phases. This finding is consistent with the XRD patterns.

AlCrFeNi was used to conduct the TEM characterization at the nanometer scale for further analyzing the eutectic microstructure, as shown in Fig. [3.](#page-3-0) The alloy shows an alternant structure with the bright and dark regions. The TEM sample has some corrosive area because of twin-jet electropolishing. The A2 and B2 phases show different corrosion resistances due to the different structures and chemical composition. The B2 phase has worse corrosion performance. The diffraction pattern of the bright region is presented in Fig. [3\(](#page-3-0)b), and that of the dark region is presented in Fig. [3](#page-3-0)(c). The bright region is B2 due to the superlattice spots presented in Fig. [3\(](#page-3-0)b). Referring to the results of EDS mapping, B2 is NiAl phase, while A2 is CrFe phase. The width of B2 is approximately 200 nm, while A2 is nearly 400 nm.

3.2 Differential Scanning Calorimeter (DSC)

DSC tests were conducted on the samples to determine the change in eutectic temperature of four alloys with different Ti contents. Figure 4 shows the DSC curves of different samples heated from room temperature to 1500 °C. The peak in the curves corresponds to the melting endothermic reaction of the materials. Ti0, Ti0.2, and Ti0.5 have an obvious endothermic peak at 1438, 1393, and 1343 °C, respectively. No other peak is found elsewhere, which means that the alloys are eutectic. By contrast, Ti1 has the main peak at 1315 °C and a small peak at 1298 \degree C. Therefore, the melting point of these intermetallic

Table 2 Compressive mechanical properties of $AICrFeNiTi_x$ at room temperature

Alloy	Yield Strength, MPa	Fracture Strength, MPa	Strain. $\%$	Density, $g/cm3$	Specific YS, $MPa \cdot cm^3/g$
AlCrFeNi	1167.45	2077.54	30.21	6.64 ± 0.04	175.83
AlCrFeNiTi _{0.2}	1556.88	1977.93	18.46	6.55 ± 0.03	237.80
AICrFeNiTi _{0.5} AlCrFeNiTi	1488.21 1416.45	1641.30 1440.43	11.32 8.28	6.42 ± 0.05 6.22 ± 0.03	231.90 227.62

Table 3 Compressive properties of some typical HEAs at room temperature

Alloy	Yield Strength, MPa	Fracture Strength, MPa	Strain, $\%$	Ref.
AlCoCrFeNi	1313.7	1943.2	19	(Ref 25)
Al0.6CoCrFeNi	\sim 400	\sim 1400	78	(Ref 36)
CoCrFeNiV	1435	1665	2.5	(Ref 37)
CoCrFeNiMnV	1660	1845	0.5	(Ref 37)
VNbMoTaW	1246	1087	17	(Ref 38)

Fig. 6 Density and specific yield strength of AlCrFeNiTi_x

compounds is similar to the temperature of the eutectic reaction. With the addition of Ti, the melting temperature (T_m) tends to diminish, which will be favorable for castability.

3.3 Mechanical Properties

Engineering compressive stress–strain curves for as-cast AlCrFeNiTi_x alloy are presented in Fig. 5 , and the results are summarized in Table [2](#page-4-0). The figure inset shows the true stressstrain curve of the samples. Table 3 shows the mechanical properties of some typical HEAs reported in the literature. The noticeable effect of Ti addition can be confirmed on the compressive behavior of the alloys. Increasing Ti content from 0 to 0.2 increases yield strength from 1167 to 1556 MPa. However, the addition of Ti decreases fracture strength from 2077 to 1977 MPa. At the same time, the maximum strain decreases from 30.21 to 18.46%. Owing to the uniform microstructure, Ti0 can distribute the deformation and stress concentration more uniformly, which can suffer much more deformation without cracking and breakage.

The atomic radius of Ti is relatively large. The lattice distortion can be expressed in the difference of atomic size (δ) .

$$
\delta = \sqrt{\sum_{i=1}^{n} c_i (1 - r_i / \overline{r})^2}
$$
 (Eq 1)

where c_i and r_i are the atom fraction and radius of element i, respectively. \overline{r} is the average radius of the elements in the alloys.

To measure the lattice distortion of the alloys, we calculate the parameters of the two phases. The parameter in AlCrFeNi is 5.60% for A2 phase, and 6.25% for B2 phase. After the addition of Ti, the parameter of atomic size mismatch for both

of the two phases increases insignificantly. The parameter in AlCrFeNiTi_{0.2} is 5.64% for A2 phase, and 6.44% for B2 phase. Based on the parameter of atomic size mismatch, we can deduce that the main reason for the increased strength in Ti0.2 is not the enhancement of lattice distortion. In the other hand, the dispersed nanoparticles in the matrix for Ti0.2 may also inhibit the migration of dislocation to increase the strength. And Ti content also affects the growth of microstructure, which increase the volume fraction of B2 phase. B2 phase has higher strength than A2 phase in general. Therefore, the addition of Ti reduces the plasticity and toughness of AlCrFeNiTi_x alloys and decreases the fracture strength and strain of $AICrFeNiTi_x$ alloys. In addition to the three factors above, brittle phase- $L2₁$ phase formed in Ti1 can result in the drop of toughness. Specific yield strength is an important index for alloys, and it is summarized in Fig. 6. As the Ti content increases, the density of the material decreases, and the specific yield strength increases constantly. The specific yield strength of all the four materials is higher than 175 MPa·cm³/g. The proposed AlCrFeNiTi_{0.2} shows superior specific yield strength at room temperature.

The fracture surface of $AICrFeNiTi_x$ was characterized by SEM, as shown in Fig. [7](#page-6-0). Figure [7\(](#page-6-0)a) shows that the fracture surface of Ti0 is relatively rough. Slip separation is observed in Fig. [7\(](#page-6-0)b), and it is one of the features of plastic fracture. The trench-like microstructure appears in Ti0.2 due to different deformation behaviors of A2 and B2. The fracture pattern belongs to ductile fracture as the dimples observed in Ti0.2. River-like patterns and tearing edges for Ti0.5 can be observed in Fig. [7\(](#page-6-0)f). Cleavage steps and obvious grain boundaries indicate that the material has a quasi-cleavage fracture, which is the reason for the sharp decrease in the strain of the material. The fracture surface of Ti1 in Fig. $7(g)$ $7(g)$ is smooth with some cracks. A large number of tearing edges indicate that the material has undergone a cleavage fracture.

3.4 Microhardness

The mechanical properties are determined by microstructure, which can be significantly affected by temperature. When the material is subjected to high-temperature environments, the decrease in material hardness may cause failure. Thus, softening resistance is important for structural materials applied in nuclear reactors. The hardness of the as-cast and annealed samples was characterized to test the softening resistance of the material at an elevated temperature. We list the average Vickers hardness taken from ten indents in Table [4.](#page-7-0) The average Vickers hardness values for as-cast Ti0, Ti0.2, Ti0.5, and Ti1 samples are 409 \pm 4, 499 \pm 4, 549 \pm 8 and 567 \pm 9 HV, respectively. And the hardness of the samples did not change significantly after the heat treatment.

Fig. 7 SEM images of fracture surface of AlCrFeNiTi_x: (a) $x = 0$, (c) $x = 0.2$, (e) $x = 0.5$, (g) $x = 1$; (b), (d), (f), and (h) are the enlarged images of (a), (c), (e), and (g), respectively

Nano-indentation is also used to measure hardness, and Nix–Gao model is used to characterize the change in material hardness with depth (Ref [39\)](#page-9-0). The model fitting formula is given as follows:

$$
H = H_0 \sqrt{1 + \frac{h^*}{h}}
$$
 (Eq 2)

where H is the hardness, H_0 is the hardness in the limit of infinite depth, h^* is the characteristic length determined by the shape of an indenter and the material, and h is the indentation depth of the material. The hardness values are inaccurate when the indented depth is less than dozens of nanometers because of the reverse indentation size effect (RISE). The hardness corresponding to a depth of less than 150 nm is discarded due to the surface effect. Figure $8(a)$ shows the relationship between hardness and depth, and Figure 8(b) presents the relationship between the reciprocal of the depth (h^{-1}) and the square of the hardness (H^2) . As shown in Fig. 8(b), the curves of Ti0 have inflection points when the depth is around 500 nm. This result indicates that the surface hardness is different from the matrix hardness of the material. According to previous reports (Ref [23](#page-8-0), [40](#page-9-0)), the EHEAs may have a large strain hardening phenomenon because of regular structure that easily stores dislocations and alternating phases. The material needs to be abraded and polished before testing, forming a hardening layer of hundreds of nanometers. Therefore, hardness varies

Table 4 Vickers Hardness and nanohardness results of as-cast and annealed AlCrFeNiTi_v

Alloy	Nanohardness, GPa	Vickers Hardness, HV
Ti ₀ (as-cast)	5.19 ± 0.16	409 ± 4
Ti0 (annealed)	5.29 ± 0.68	411 ± 7
$Ti0.2$ (as-cast)	6.86 ± 0.22	499 ± 4
Ti0.2 (annealed)	7.26 ± 0.23	526 ± 9
$Ti0.5$ (as-cast)	7.10 ± 0.27	549 ± 8
Ti0.5 (annealed)	7.28 ± 0.20	550 ± 3
Ti1 (as-cast)	7.69 ± 0.45	567 ± 9
Ti1 (annealed)	8.02 ± 0.29	570 ± 4

from the surface toward the core. The nanohardness of the material is summarized in Table 4. The hardness is 5.19 GPa without Ti addition. By contrast, the hardness increases significantly after Ti addition, and it is higher than 7 GPa, maybe because Ti promotes the lattice distortion of the alloy and intermetallic compounds form in Ti1. However, the hardness of the material does not change significantly after the high-temperature treatment at 500 $^{\circ}$ C. This finding means that the material has good softening resistance at a high temperature.

4. Summary

In this study, AICrFeNiTi_x (x=0, 0.2, 0.5, 1) was synthesized by cold crucible levitation melting. The effect of Ti content on the phase structure, microstructure, and mechanical properties of AlCrFeNiTix was analyzed systematically. The major conclusions of this study are drawn as follows:

- 1. The XRD showed ordered BCC and disordered BCC in the three types of alloys. Furthermore, $L2₁$ phases were formed in Ti1. The feature of the eutectic structure was found from SEM images, which was identified to be the combination of the B2 phase (Ni, Al) and the A2 phase (Fe, Cr). Nanosized precipitates could be observed inside the B2 phase with the Ti addition.
- 2. With the addition of Ti, the yield strength of AlCrFeNi- Ti_x alloys increased from 1167 MPa (Ti0) to 1556 MPa (Ti0.2). However, the toughness of AlCrFeNiTi_x alloys decreased at the same time. Due to the low density of alloys $(< 7 \text{ g/cm}^3)$, alloys can be defined as lightweight alloys. The addition of Ti decreased the density and increased the specific yield strength of AICrFeNiTi_x alloys. The specific yield strength of Ti0.2 even reached 237 MPa·cm³/g.
- 3. The hardness of the alloys increased with Ti addition. After high-temperature annealing, the hardness of AlCr-FeNiTix alloys changed insignificantly, which meant that the material had great softening resistance at a high temperature (500 \degree C).

Fig. 8 (a) Average nanohardness versus the indentation depth of as-cast and annealed AlCrFeNiTi_x; (b) Curves of H^2-h^{-1} for average nanohardness of the alloy

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