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High-throughput determination of mechanical and diffusion properties of Ti–Ta–Fe alloys

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Abstract: The mechanical and diffusion properties of Ti−Ta−Fe alloys in the Ti-rich region were investigated by utilizing a high-throughput method, with the combination of nanoindentation and diffusion couple techniques. Five groups of ternary Ti−Ta−Fe diffusion couples were prepared after annealing at 1273 K for 25 h. The composition-dependent mechanical properties of bcc Ti−Ta−Fe system were experimentally determined by means of nanoindentation and electron probe microanalysis (EPMA) techniques. Moreover, the interdiffusion coefficients of Ti−Ta−Fe alloys at 1273 K were confirmed from the composition gradients of the ternary diffusion couples with the support of a pragmatic numerical inverse method. A composition-dependent database on the mechanical and diffusion properties of Ti−Ta−Fe alloys was carefully established and utilized for the discussion of the processability during the hot working. The results indicated that the content of Fe should be controlled for the Ti alloys with high hardness and low Young's modulus.

Key words: Ti−Ta−Fe alloys; diffusion couple; nanoindentation; interdiffusion coefficients; mechanical properties

1 Introduction

Due to superior mechanical properties including low Young's modulus and high specific strength, Ti alloys containing Ta have attracted more and more attention [1−3]. Ti−Ta-based alloys are regarded as one of the potential biomedical titanium alloys, but the expensive cost extremely restricts their applications. In order to overcome the drawback, cheaper alloying metal like Fe is more suitable to be added into Ti−Ta alloys. Moreover, the addition of Fe can stabilize β(bcc) phase of Ti alloys, resulting in the improvement of the mechanical properties [4]. However, systematic studies on the composition-dependent mechanical and diffusion properties in the Ti-rich Ti−Ta−Fe system are still missing. A deep understanding on the mechanical and diffusion properties of the Ti−Ta−Fe alloys is necessary to be pursued for the design of novel bio-alloys.

Young's modulus, hardness and interdiffusion coefficient refer to the important mechanical and diffusion properties of the materials, which are necessarily considered during the design of the novel metallic implant biomaterials [5–8]. The traditional method, using repeated compositional adjustments and experimental testing, merely provides the limited experimental data, which seriously blocks the development of the mechanical property database. For the sake of much more experimental Young's moduli and hardness of the alloys, a high-throughput method through the combination of the nanoindentation tests with the

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diffusion couple technique was proposed [9] and conducted for several Ti−Nb−Zr-based alloy systems [10−15]. In addition, interdiffusion coefficients can be also obtained from the composition gradient of diffusion couples via a pragmatic numerical inverse method [16−18]. Considering that they are related to the steady-state rate at the intermediate temperature, the diffusion information and Young's modulus are generally used to judge the difficulty of processing [14].

In this work, the combination of diffusion couple, field emission electron probe microanalysis (Fe-EPMA), and nanoindentation techniques are utilized to investigate the mechanical and diffusion properties in the single bcc phase of Ti-rich Ti−Ta−Fe system over a wide composition range at 1273 K. Moreover, a pragmatic numerical inverse method is used to determine the interdiffusion coefficients of the Ti−Ta−Fe alloys at 1273 K. And then, the composition-dependent mechanical and diffusion properties of Ti-rich Ti−Ta−Fe alloys are discussed.

2 Experimental

2.1 Material preparation and characterization

The slugs of Fe (purity: 99.95 wt.%), Ta (purity: 99.95 wt.%), and Ti (purity: 99.999 wt.%) were used for the fabrication of the Ti−Ta−Fe diffusion couples. Figure 1(a) shows the isothermal section of the Ti-rich Ti−Ta−Fe system at 1273 K calculated from the thermodynamic parameters [4]. Based on the calculated isothermal section, the binary and ternary alloys in the single bcc phase zone of Ti−Ta−Fe system were designed, and the compositions of couples C1−C5 in the Ti-rich Ti−Ta−Fe system were presented in Table 1. Specimens prepared by arc melting under an Ar atmosphere using a non-reactive W electrode (WKDHL-1, Opto-electronics Co., Ltd., Beijing, China) were cut into blocks with the size of 7 mm × 7 mm × 2 mm. Being sealed into vacuum quartz tubes, the ground blocks were homogenized at (1273±2) K for 7 d. After the homogenization annealing, the ground blocks with any two components were bound by Mo clamps, sealed into vacuum quartz tubes, and annealed at (1273±2) K for 25 h in an ELF1106-type furnace (Carbolite Gero Co., Ltd., United Kingdom). After annealing, the Ti−Ta−Fe diffusion couples were quenched in

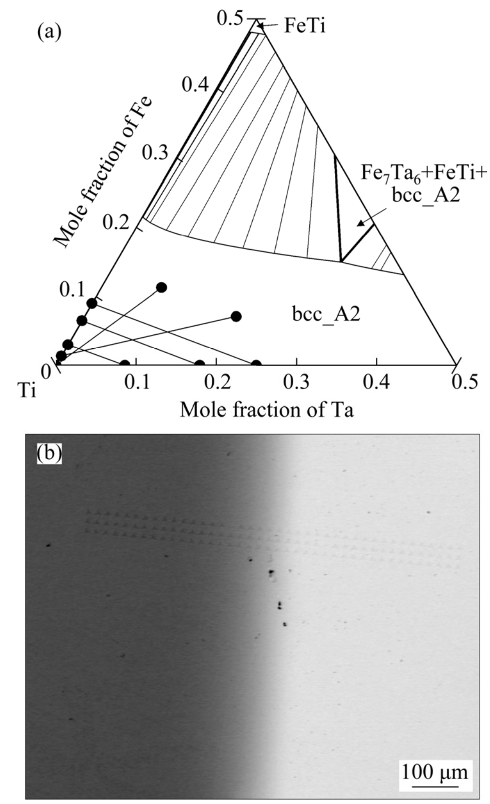

Fig. 1 Calculated isothermal section at 1273 K in Ti-rich Ti−Ta−Fe system by using thermodynamic parameters available in Ref. [4] (a), and backscattered electron image of Ti−1.36Fe/Ti−18.95Ta−7.10Fe diffusion couple (b)

Table 1 Alloy compositions for Ti−Ta−Fe diffusion couples

Couple	Composition/at.%
C1	Ti−3.00Fe/Ti−8.60Ta
C2	Ti−6.50Fe/Ti−17.95Ta
C3	Ti−9.00Fe/Ti−25.00Ta
C4	Ti−1.36Fe/Ti−18.95Ta−7.10Fe
C5	Ti/Ti−7.56Ta−11.25Fe

the flowing water. Cross-sections of the diffusion couples were ground and polished by SiC papers and diamond pastes, respectively. The compositions along the composition gradient direction of 5 ternary Ti−Ta−Fe diffusion couples were determined with the support of Fe-EPMA (JXA–8530, JEOL, Japan), and the composition resolution for each component was ±0.5 at.% in the measurement of EPMA. Young's moduli (E) and hardness (H) were measured by the nanoindenter (Keysight G200, Agilent Technology, USA) with an in-depth control (2000 nm) according to the Oliver-Pharr analysis [19]. Moreover, the corresponding energy was obtained from the load−displacement curve, and ratio of the elastic indentation energy to the total deformation energy (U_{el}/U_{tot}) was calculated as the elastic recovery [14].

2.2 Determination of interdiffusion coefficients in alloys

Based on the Fick's second law, a pragmatic numerical inverse method was proposed to determine the interdiffusion coefficients along with the composition variations of the binary or multicomponent diffusion couples [16,17]. The composition-dependent interdiffusion coefficients of Ti-rich Ti−Ta−Fe system were calculated with the support of a high-throughput determination of interdiffusion coefficients (HitDIC) code [20]. In the present work, the thermodynamic parameters presented in Ref. [4] were utilized as well as the mobility parameters reported in Refs. [21,22].

3 Results and discussion

3.1 Mechanical and wear properties of Ti−Ta−Fe alloys

Considering that all the annealed diffusion couples are located in the single bcc phase region, one typical microstructure is given in Fig. 1(b), which shows the backscattered electron image of C4 annealed at 1273 K for 25 h. Using the contact boundary of the end-members as the reference position, mechanical properties including Young's modulus, hardness and elastic recovery as a function of composition can be obtained by correlating the fitted composition vs. position curves with the nanoindentation results vs. position, which has been performed in Ref. [23]. Figure 2 illustrates the composition-dependent mechanical properties (i.e., Young's modulus, hardness, and elastic recovery) in the Ti−Ta−Fe diffusion couples, accompanied with the corresponding experimental errors. Obviously, the measured mechanical properties are composition-dependent. As shown in Fig. 2, the variations of the experimental Young's moduli, hardness and elastic recovery are within the range of 88−165 GPa, 3.2−7.2 GPa and 0.18−0.36, respectively. It can be seen from Figs. 2(a′−c′) that the experimental Young's moduli and hardness from the side of Ti−Fe rapidly decrease to those of Ti−Ta, while the elastic recovery doesn't vary very significantly. The experimental hardness and elastic recovery variations from Ti−Fe to Ti−Ta−Fe in Fig. 2(d, d′) are similar to the trends in Fig. 2(e, e′), which show a maximum value at the interface of the couples. In Figs. 2(d, d′, e, e′), the Young's moduli of the bcc Ti alloys decrease and then increase from the side of Ti−Fe to the side of Ti−Ta−Fe.

In general, high wear resistance can prolong the service life of the materials [24,25]. According to the wear theory [26], wear resistance can be indirectly represented by using the ratios of hardness to Young's modulus including H/E and H^3/E^2, which separately symbol the ability to resist elastic deformation and plastic deformation in loaded contact. Based on the above-mentioned Young's modulus and hardness, these two ratios are presented to evaluate the wear resistance of the bcc-Ti alloys, and the calculations are presented in Fig. 3. In the figure, the variations of H/E and H^3/E^2 of the Ti alloys in the Ti−Ta−Fe diffusion couples are similar to each other. The values 0.04 and 0.009 GPa recommended in Ref. [12] are used as the reference states of H/E and H^3/E^2 for the comparison. In Fig. 3, the H/E ratio of Ti alloys with high concentrations of Ta and Fe is larger than 0.04, while the H^3/E^2 value of most composition points is less than 0.009 GPa. The result indicates that Ti−Ta−Fe alloys have a certain ability to resist elastic deformation, but limit wear resistance during the plastic deformation.

It can be seen that the experimental database of Young's modulus, hardness, and indirect wear resistance parameter corresponding to the different alloy compositions (200 points) was built for the Ti alloys in the bcc Ti−Ta−Fe system using the diffusion couple technique. To screen the bio-Ti alloy with desired mechanical properties, a five-dimensional scatter plot of the data points is shown in Fig. 4, in which the X, Y and Z axes seperately represent the Fe content, Ta content and Young's modulus, while the color scale and size of symbols are determined by hardness and indirect wear resistance, respectively. It is noted that the point projections of the measured data into a two-dimensional plane (solid circles in orange, aqua and pink colors) are also presented in Fig. 4, which can enable us to assess the exact values of alloy compositions and Young's moduli.

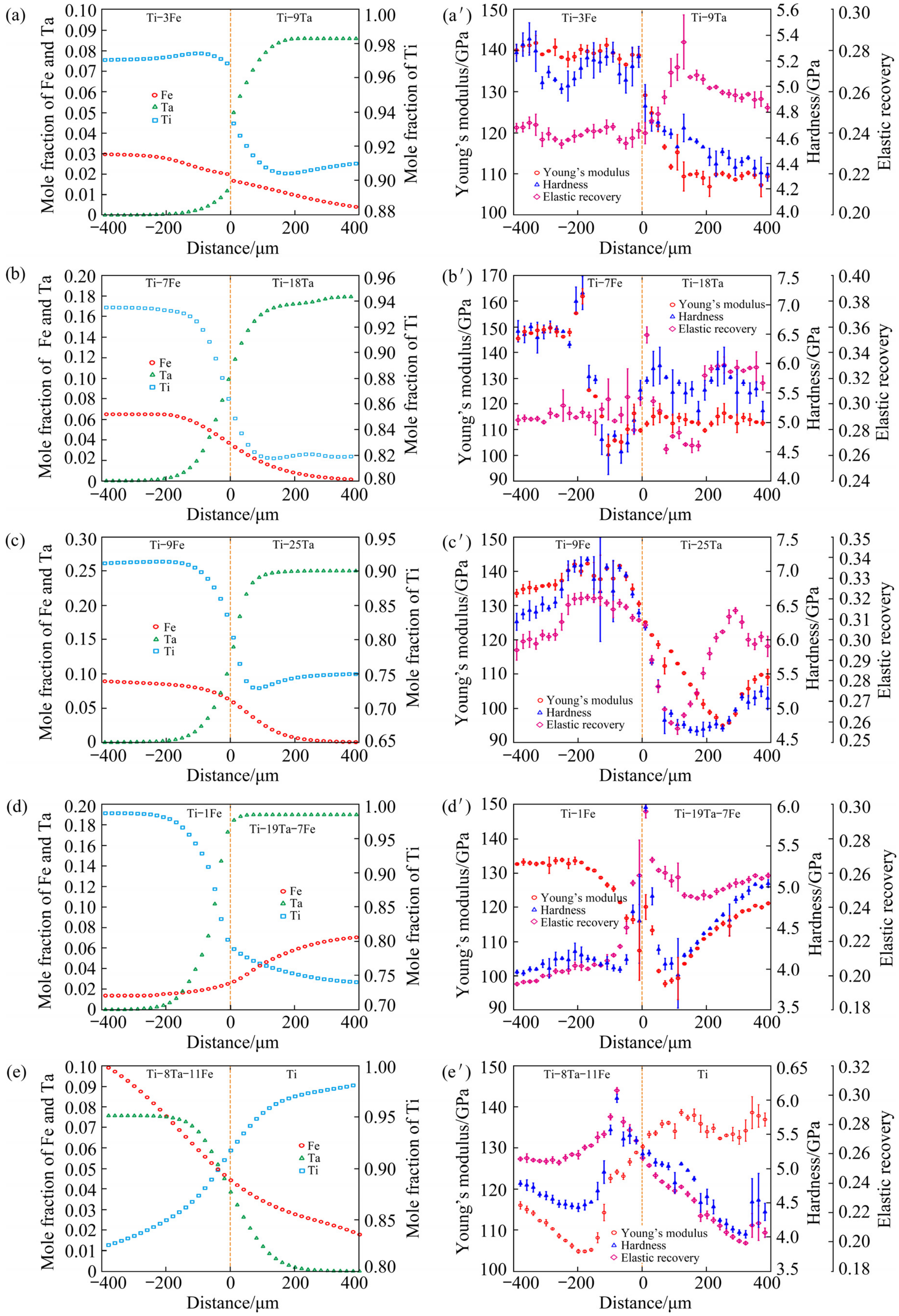

Fig. 2 Experimental composition profiles (a, b, c, d, e), and Young's modulus, hardness and elastic recovery (a′, b′, c′, d′, e′) of bcc Ti-based alloys in couples of C1−C5 annealed at 1273 K for 25 h: (a, a′) C1; (b, b′) C2; (c, c′) C3; (d, d′) C4; (e, e′) C5

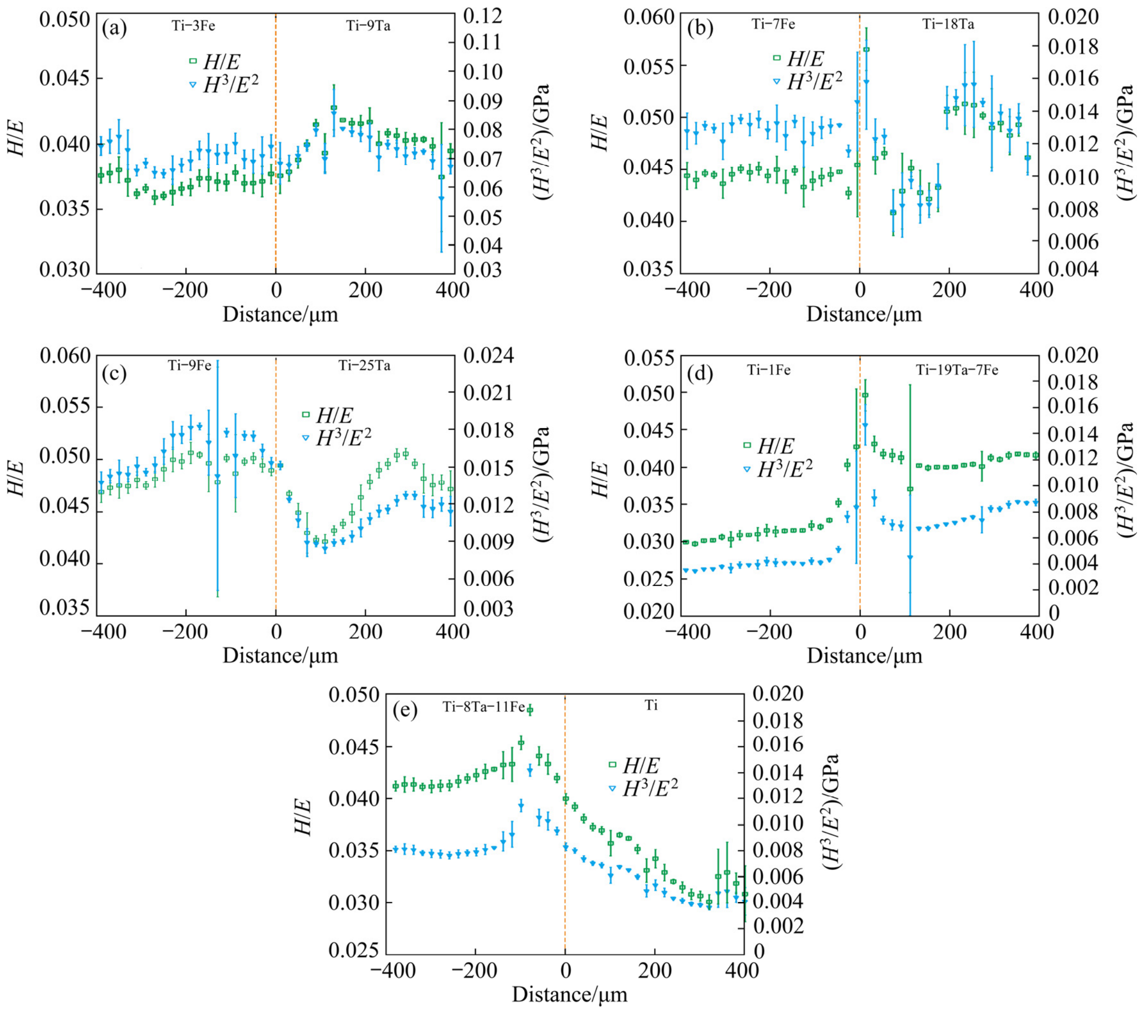

Fig. 3 Ratios H/E and H^3/E^2 of bcc Ti-based alloys in C1−C5 diffusion couples annealed at 1273 K for 25 h: (a) C1; (b) C2; (c) C3; (d) C4; (e) C5

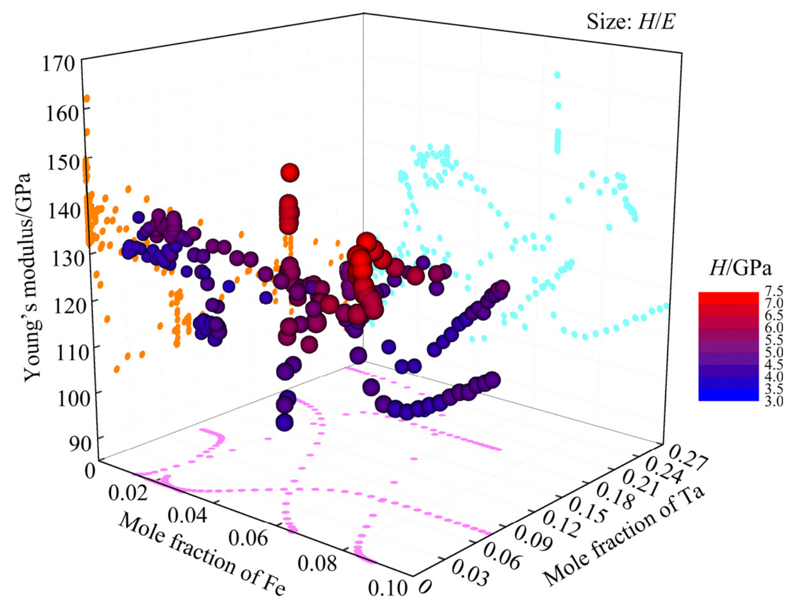

Fig. 4 Relationship among alloy composition, Young's modulus, hardness, and H/E of bcc Ti-based alloys in Ti−Ta−Fe diffusion couples

3.2 Diffusion properties of Ti−Ta−Fe alloys

Figure 5 displays the model-predicted composition profiles of Ti−Ta−Fe diffusion couples amealed at 1273 K, compared with the measured data. Most of the experimental profiles are in good agreement with the model-predicted composition profiles, meaning that the modeling parameters are reliable and can be utilized to provide the accurate interdiffusion coefficients. The constructed inter-diffusion coefficients of Ti-rich Ti−Ta−Fe system at 1273 K are demonstrated by using the color variations in the composition space of a ternary system in Fig. 6. The figure shows that the main and cross interdiffusion coefficients using pragmatic numerical inverse method varied with the contents of elements Ta and Fe. Main interdiffusion coefficients, $\tilde{D}^{\mathrm{Ti}}_{\mathrm{FeFe}}$ and $\tilde{D}^{\mathrm{Ti}}_{\mathrm{TaTa}}$, are positive, while both the cross interdiffusion coefficients, $\tilde{D}^{\mathrm{Ti}}_{\mathrm{FeTa}}$ and $\tilde{D}^{\mathrm{Ti}}_{\mathrm{TaFe}}$, are negative. The main interdiffusion coefficient $\tilde{D}^{\mathrm{Ti}}_{\mathrm{TaTa}}$ decreases mainly with the increasing content of Ta in the Ti–Ta–Fe system, and the other main one $\tilde{D}^{\mathrm{Ti}}_{\mathrm{FeFe}}$ decreases with the increasing Fe and Ta contents. In addition, the cross interdiffusion coefficients show complex variations with the contents of Ta and Fe.

3.3 Hot processability prediction of Ti−Ta−Fe alloys

Recently, the ratio of effective diffusion coefficient to cube of Young's modulus (D_{eff}/E^3)

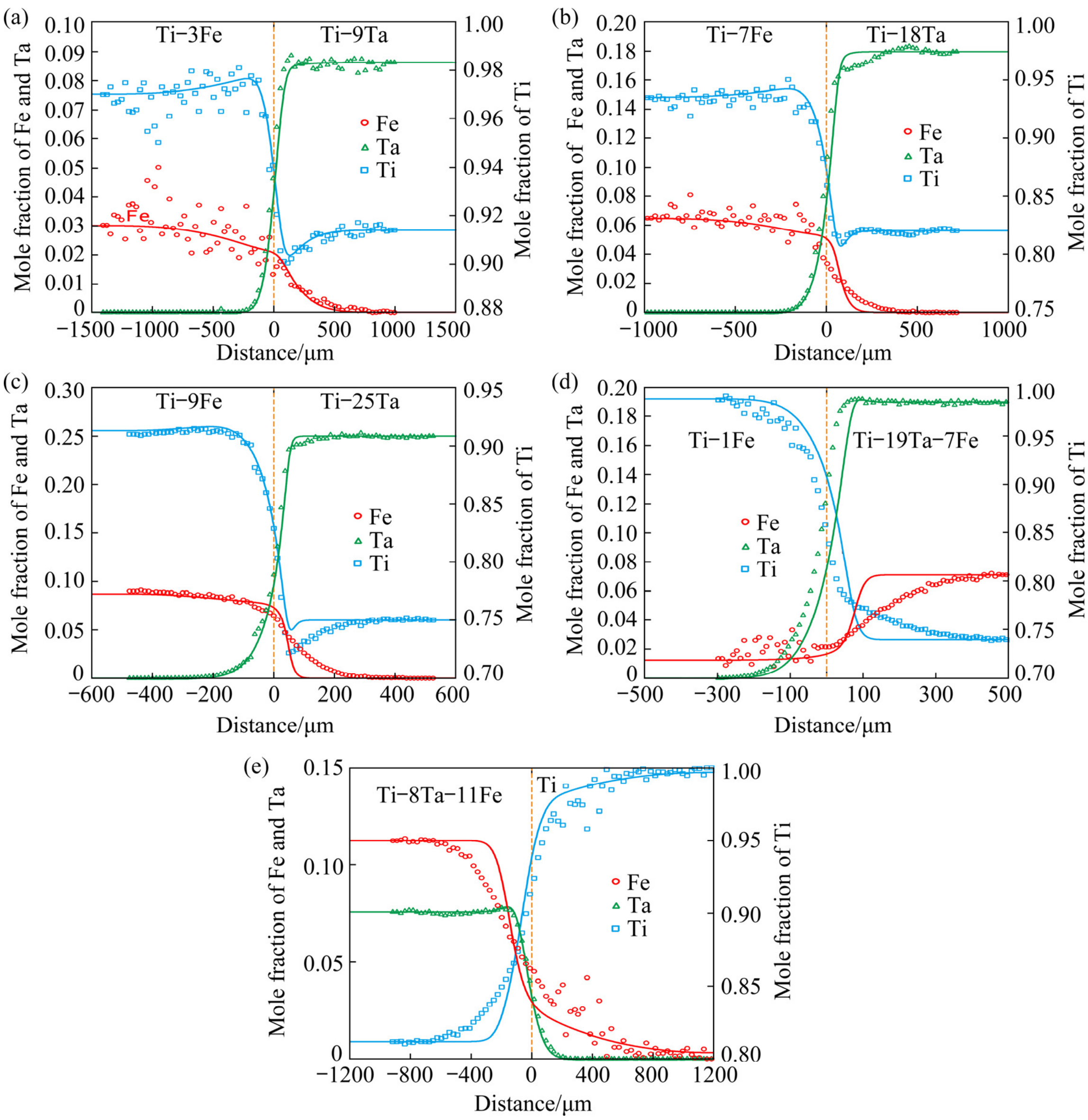

Fig. 5 Measured and model-predicated composition profiles in couples of C1−C5 annealed at 1273 K for 25 h: (a) C1; (b) C2; (c) C3; (d) C4; (e) C5

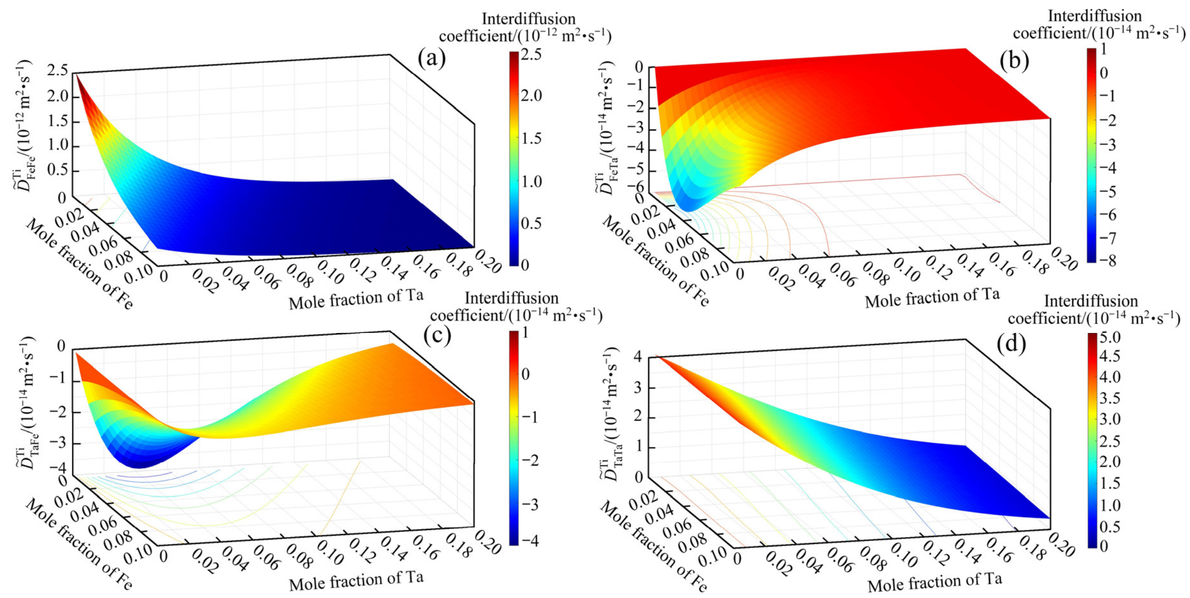

Fig. 6 Main and cross interdiffusion coefficients of Ti-rich Ti−Ta−Fe system at 1273 K determined by using pragmatic numerical inverse method

was proposed as a hot workability parameter to characterize the processability during the hot working [13]. It is noted that the effective diffusion coefficient is equivalent to the main interdiffusion coefficient of the solute element with large atomic radius, which is consistent with the experimental investigations in Ref. [27]. The hot workability parameter D_{eff}/E^3 at each composition point of Ti−Ta−Fe diffusion couples can be obtained from the experimental Young's moduli and the main interdiffusion coefficients at 1273 K, which is shown in Fig. 7. Due to the atomic radii of two solute elements Ta and Fe, the effective diffusion coefficient D_{eff} of Ti−Ta(−Fe) and Ti−Fe alloys and pure Ti are the interdiffusion coefficients $\tilde{D}^{Ti}_{TaTa}$ and $\tilde{D}^{Ti}_{FeFe}$, and self-diffusion coefficient D^{Ti}_{Ti}, respectively. In Ref. [13], a value of 7.25×10^{-20} m^2·s^{-1}·GPa^{-3} is recommended as the reference of Ti−6Al−4V alloy for the comparison. In Fig. 7, the hot workability parameters of Ti−Fe alloys are larger than the recommended value, but those of other Ti alloys in the Ti−Ta−Fe diffusion couples are lower than the recommended value, which is caused by the low interdiffusion coefficients in Ti alloys containing the element Ta. Moreover, the hot workability parameter of Ti−Fe alloys decreases with the increasing Fe content. The results indicate that the addition of Ta and high content of solute element Fe are detrimental to the processability of Ti alloys at intermediate temperatures. In order to improve the processability, element Zr with larger atomic radii and higher diffusion coefficient can be added into Ta-containing Ti alloys according to the recent investigations [13,15].

4 Conclusions

(1) The experimental mechanical properties of the bcc Ti−Ta−Fe alloys are composition-dependent, which are urgently needed for the alloy design. The experimental Young's moduli, hardness and elastic recovery of the Ti−Ta−Fe alloys are in the range of 88−165 GPa, 3.2−7.2 GPa and 0.18−0.36, respectively. The increasing content of Fe brings in the simultaneous increase of hardness and Young's modulus. Thus, the content of Fe should be controlled to obtain the Ti alloys with low Young's modulus.

(2) The high H/E ratio and low H^3/E^2 ratio indicate that Ti–Ta–Fe alloys have high wear resistance during the elastic deformation, but limit wear resistance during the plastic deformation. This result is caused by the fact that the increment of Young's modulus with the contents of Ta and Fe is obviously more than that of hardness in the Ti-rich Ti−Ta−Fe system. The plot of mechanical and wear properties for Ti−Ta−Fe alloys over a wide composition range is presented.

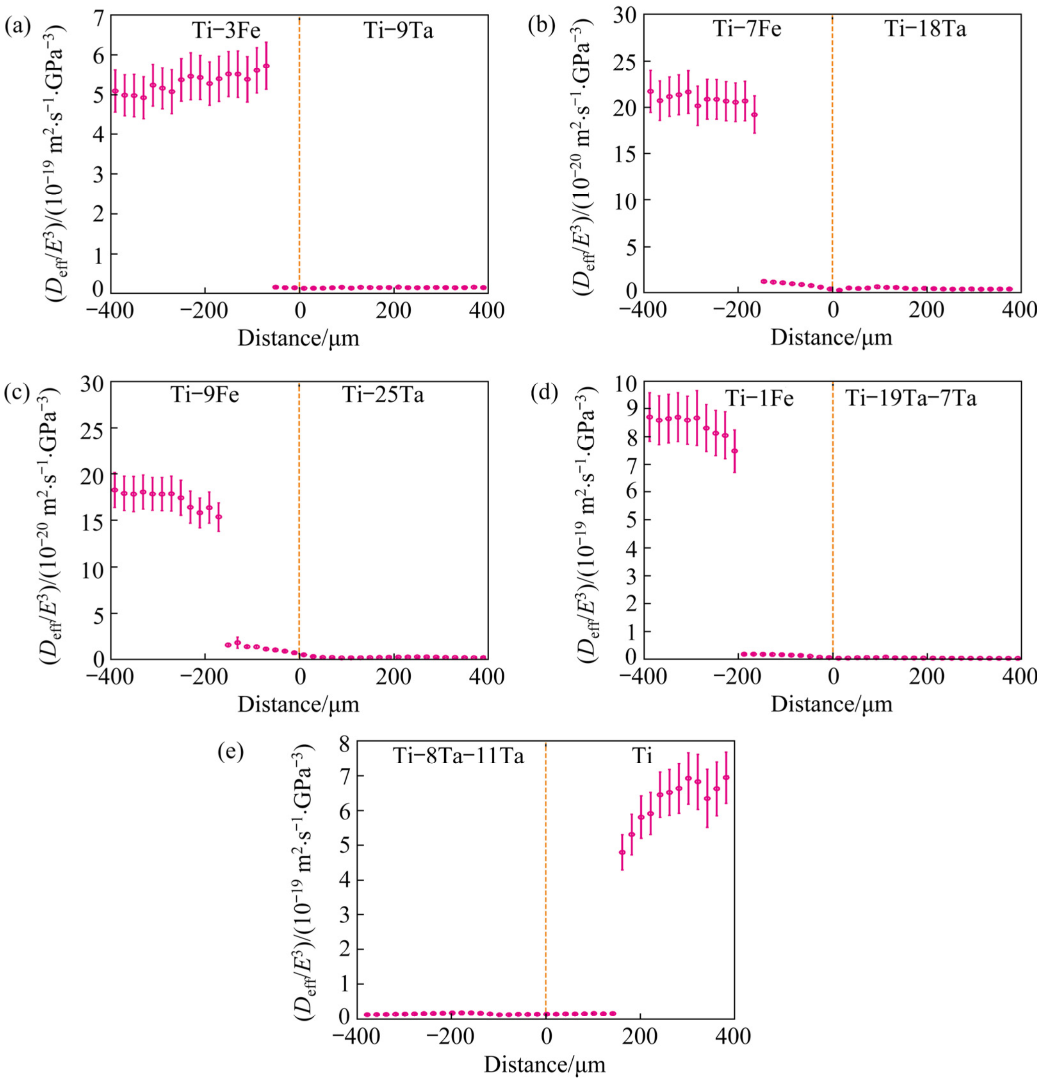

Fig. 7 Hot workability parameter of bcc Ti-based alloys in C1−C5 diffusion couples annealed at 1273 K for 25 h: (a) C1; (b) C2; (c) C3; (d) C4; (e) C5

(3) The composition-dependent interdiffusion coefficients of the bcc Ti−Ta−Fe alloys are determined by using the pragmatic numerical inverse method from the experimental composition profiles of ternary diffusion couples. A plot of a hot workability parameter in the Ti-rich Ti−Ta−Fe system is presented. This work reveals that high content of the solute element Ta is unfavourable for the processability during the hot working. Elements with large atomic radii and high diffusion coefficient need to be added in the Ti alloys for the nice hot processability.

Acknowledgments

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Ti−Ta−Fe 合金力学和扩散性能的高通量测定

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摘 要：结合纳米压痕和扩散偶方法对 Ti−Ta−Fe 合金富 Ti 端的力学性能和扩散性能开展高通量研究。通过在 1273 K 固溶退火 25 h 制备 5 组三元 Ti−Ta−Fe 扩散偶。借助纳米压痕和电子探针等技术确定 Ti−Ta−Fe 体系体心立方相随成分变化的力学性能。利用实用高效数值回归方法从三元扩散偶的成分梯度中获取 1273 K 温度下的互扩散系数。基于以上实验结果构建一套 Ti−Ta−Fe 合金随成分变化的力学和扩散性能数据库。该数据库可用于钛合金热加工过程中的加工变形性能分析。结果表明，控制 Fe 含量可提高钛合金的硬度和降低杨氏模量。

关键词：Ti−Ta−Fe 合金；扩散偶；纳米压痕；互扩散系数；力学性能

(Edited by Xiang-qun LI)