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Deuterium permeability of a novel AlCrTaTiZr high entropy alloy coating

Wei Zhang †, Long Wang ‡, Xiaofang Luo §, Baoping Gong †, Xiaoyu Wang †, Jijun Yang ‡ and Yongjin Feng ‡

1 Key Laboratory of Radiation Physics and Technology of Ministry of Education, Institute of Nuclear Science and Technology, Sichuan University, Chengdu, 610064, People’s Republic of China
2 Southwestern Institute of Physics, Chengdu 610225, People’s Republic of China
* Authors to whom any correspondence should be addressed.
E-mail: wanglong@swip.ac.cn and jjyang@scu.edu.cn

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Abstract

AlCrTaTiZr, AlCrTaTiZr nitride, and AlCrTaTiZr oxide coatings as tritium permeation barriers (TPBs) were prepared by magnetron co-sputtering technology on a CLF-1 (RAFM) substrate, to evaluate their hydrogen/deuterium permeation resistance. X-ray diffraction (XRD) analysis showed that the as-deposited coatings were amorphous. Scanning electron microscopy (SEM) results revealed that the deposition rates of the AlCrTaTiZr coating, AlCrTaTiZr nitride coating and AlCrTaTiZr oxide coating were approximately 2 μm h⁻¹, 0.75 μm h⁻¹ and 0.15 μm h⁻¹, respectively. The properties of these high-entropy alloy (HEA) coatings were studied with electrochemical hydrogen (ECH) permeation test equipment and a high-temperature gas-driven deuterium permeation (GDP) device. The AlCrTaTiZr HEA coating had the best hydrogen permeation resistance during the electrochemical hydrogen permeation test. However, the AlCrTaTiZr nitride coating had the best deuterium permeation resistance during the gas–deuterium permeation test. Annealing experiments of these coatings showed that changes in their microstructures during the gas-driven deuterium permeation test were the main reasons for the different permeabilities of these coatings during the heating and cooling processes. The oxidization of the HEA coating during the deuterium permeation experiment may have been the main reason that the HEA coating showed better deuterium permeation resistance than the HEA oxide coating.

1. Introduction

As possible permanent energy for the future of humankind, fusion reactors have attracted much attention from researchers interested in their development in recent decades [1]. A tritium breeding blanket is necessary for a D-T fusion reactor, through which tritium breeding, radiation shielding and heat output conversion proceed [2]. The tritium breeding blanket includes a tritium breeder, neutron multiplier and structural materials [3]. The structural materials in the tritium breeding blanket face severe neutron radiation exposure [4] by the tritium breeder and thus corrode [5-8]. In addition, as a radioactive isotope of hydrogen, tritium exhibits very high permeability because of its small mass and size [9]. Tritium permeation through structural materials is a critical issue, and uncontrolled tritium and its isotopes affect the self-sustainment of the fusion reaction, cause hydrogen embrittlement of structural materials and produce radiological health hazards [10]. Therefore, it is crucial to mitigate tritium permeation to a proper level for the development of fusion reactors.

One of the main strategies to control tritium migration through structural materials is in situ oxidization by controlling the oxygen concentration to be in a range suitable for forming a stable oxide layer [11]. However, it is difficult to maintain the targeted oxygen level in a complex heat-transport system, and an excessive oxidation temperature also seriously affects the performance of structural materials. Alternatively, a thin ceramic coating as a tritium permeation barrier (TPB) on the surface of structural materials has been recently proposed to
minimize tritium permeation [9, 12]. Several ceramic coatings have been investigated, such as Al$_2$O$_3$ [13–15], Cr$_2$O$_3$ [16], Y$_2$O$_3$ [17], Er$_2$O$_3$ [18], SiC, TiC and TiN coatings [2, 10]. Among these coatings, Al$_2$O$_3$ coatings, particularly the α-Al$_2$O$_3$ coating, is considered to be among the most promising tritium barrier coatings due to their excellent corrosion resistance, high-dose irradiation tolerance, effective hydrogen isotopic permeation reduction and unprecedented mechanical properties [13, 15, 19]. However, the preparation temperature of α-Al$_2$O$_3$ coatings exceeds 1000°C, and this high temperature profoundly reduces the performance and applicable life of structural materials.

High-entropy alloys (HEAs), as new alloy systems based on from five to ten elements in equal or near-equal atomic ratios, were first proposed in 2004 by Yeh et al. [20–24]. In recent years, HEAs have attracted much attention due to their high strength and hardness [25], excellent corrosion resistance [23, 26, 27], high phase stability [28] and excellent irradiation resistance [29]. Similarly, HEA-based coatings have attracted considerable attention for use in surface engineering applications such as diffusion barriers and corrosion barriers [30]. For example, Dou et al. [26] prepared FeAlCoCuNiV high-entropy alloy coatings by direct current magnetron sputtering, and the results showed that the hardness and Young’s modulus of the HEA coatings were 19.7 and 189 GPa, respectively. All coatings exhibited better electrochemical corrosion resistance than stainless steel in acidic alkali and salt corrosion media. Chen et al. [31] pointed out that the as-deposited Cr$_{0.26}$Cu$_{0.3}$Fe$_{0.1}$Mo$_{0.15}$Ni$_{0.19}$ HEA coating had superior hardness (12.5 GPa) and high-temperature corrosion resistance relative to other coatings. Li et al. [27] revealed that FeAlCuCrCoMn HEA coatings exhibited better corrosion resistance than 201 stainless steel in acidic alkali and salt corrosion media. All of these excellent properties make HEA coatings appealing for use in tritium permeation barriers (TPBs) of structural materials in tritium breeding blankets.

In this study, a novel type of AlCrTaTiZr HEA coating was prepared by magnetron co-sputtering technology on a CLF-1 substrate. The morphology and microstructure of the HEA coatings were studied in detail. Electrochemical hydrogen permeation and gas-driven deuterium permeation were performed to analyse the hydrogen/deuterium permeation resistance of these HEA coatings.

2. Experimental procedure

2.1. Coating preparation

AlCrTaTiZr HEA coatings were prepared by the magnetron co-sputtering method on Si and CLF-1 substrates. The CLF-1 substrates were cut into pieces with a 29 mm diameter and 0.5 mm thickness for use in electrochemical hydrogen permeation tests, and pieces with a 12 mm diameter and 0.5 mm thickness for use in gas-driven deuterium permeation tests. The nominal chemical composition (by weight) of the CLF-1 substrate was 8.5% Cr, 1.5% W, 0.3% V, 0.5% Mn, 0.08% Ta, and the balance consisted of Fe. Before the coatings were prepared, the CLF-1 substrates were polished to a mirror finish by using different grit water sandpapers and 1.5 μm diamond pastes. The polished samples were cleaned at 80°C in ethanol, acetone, and deionized water. HEA coatings were deposited by a magnetron sputtering device (QX-500 ultrahigh vacuum magnetron sputtering coating equipment, Chengdu Qixing Vacuum Coating Technology Co., Ltd, China) with metal targets of Al, Cr, Ta, Ti and Zr with purities of 99.999% in Ar, Ar + O$_2$ and Ar + N$_2$ atmospheres. During the coating preparation process, pre-sputtering of Ar was performed to remove the surface oxide layers from the target. The distance between the sample holder and metal targets was maintained at 9 cm. The background pressure was set as 8.0 × 10$^{-4}$ Pa before Ar gas was introduced. The Ar gas flow was set at 48 standard cubic centimetre per minute (sccm), and the O$_2$ and N$_2$ flows were set to 8 sccm and 10 sccm to obtain oxides and nitrides, respectively. The sputtering deposition power was 200 W and the sputtering time was 2 h for the HEA, HEA oxide and HEA nitride coatings to ensure a certain thickness of high-quality coating. Annealing treatment of the HEA coatings was performed at 700°C for 1 h in an Ar gas atmosphere.

2.2. Characterization

The phase structure of the coatings was analysed by grazing incidence X-ray diffraction (GIXRD, Empyrean, PANalytical) with an incidence angle of 1°. The microstructure and chemical composition of the samples were characterized by a field emission scanning electron microscopy (FESEM, S-4800, Hitachi, Japan).

2.3. Hydrogen permeation experiment

The electrochemical hydrogen permeation experiment was performed using the Devanathan and Stachurski double electrolyzer model [32]. A sample was sandwiched between the cathode chamber and anode chamber, and 0.2 mol l$^{-1}$ KOH was used as the electrolyte in both the cathode chamber and anode chamber. With increasing test time, the recorded current density increased to a steady-state value. The hydrogen permeation
resistance was evaluated by comparing the difference between the steady-state current density and the initial point of the hydrogen charge.

### 2.4. Deuterium permeation experiments

As one of the most important performance indexes of the tritium permeation barrier (TPB), gas-driven deuterium permeation tests were used to analyse the deuterium permeation resistance of the HEA coatings. Figure 1 presents a diagram showing the deuterium gas-driven permeation device at the Southwestern Institute of Physics (SWIP). The deuterium gas-driven permeation equipment is separated by the sample, including upstream and downstream. The upstream region is used for the gas supply and the downstream region is used for permeation signal monitoring. In the upstream, a dynamic vacuum ($10^{-5}$ Pa) was achieved through a turbomolecular pump. At the same time, the dynamic vacuum ($10^{-7}$ Pa) of the downstream was also achieved through a turbomolecular pump and ion pump. During the experimental process, the deuterium pressure was maintained at a constant pressure of 70 kPa, and the pressure was monitored by a capacitance diaphragm gauge. The temperature of the sample was controlled from 550 ℃ to 700 ℃ using a muffle furnace. A quadrupole mass spectrometer (QMS) was used to track the ion current of D$_2$ (mass 4) at the downstream. Before the measurement was taken, the D$_2$ signal was calibrated using a D$_2$ standard leak to determine the relationship between leakage and ion current.

The steady-state deuterium permeation flux per unit area through a sample is determined by the following equation [33–36]:

$$J = \frac{P \cdot p^n}{d}$$

Where $J$ is the permeation flux, $P$ is defined as the permeability of the material, $p$ is the driving pressure, $d$ is the sample thickness and $n$ is the pressure exponent, which represents the permeation regime: a value of 0.5 indicates that the rate-limiting process is the diffusion of hydrogen isotope atoms, and a value of 1 indicates that the rate-limiting process is dominated by molecular reactions such as adsorption and recombination at the sample surface.

### 3. Results and discussion

#### 3.1. Microstructure and hydrogen/deuterium permeation resistance

Figures 2(a)–(c) presents the SEM images showing the surface morphology of the HEA coatings, revealing that the coatings had a uniform, dense and compact surface morphology. No cracks were observed in the coatings, indicating that the coatings were deposited into a high-quality coat. In addition, the chemical composition of the as-deposited coating revealed that a near equimolar ratio HEA coating was successfully prepared, which corresponded to the definition of HEAs formed by mixing five or more metallic elements with equimolar or near equimolar ratios, and for which the concentration of each primary element can vary between 5 and 35 atomic % [24]. The thicknesses of the HEA, HEA nitride and HEA oxide coatings were approximately 4 μm, 1.5 μm and 0.3 μm, respectively, and the corresponding deposition rates were 2 μm h$^{-1}$, 0.75 μm h$^{-1}$ and 0.15 μm h$^{-1}$, respectively, as shown in figures 2(d)–(f). There were no obvious defects in the cross-sections of the coatings, indicating a good interfacial bond strength and confirming high quality deposition. In addition, an obvious
columnar crystal structure was observed in the cross-sections of the coatings. The columnar crystals penetrating the entire coating cause deuterium to reach the substrate surface directly along the grain boundary, which seriously degrades the hydrogen/deuterium permeation resistance of the coating [37–39].

Figure 3 shows the XRD patterns of the as-deposited AlCrTaTiZr based HEA coatings. As the picture reveals, the AlCrTaTiZr coating exhibited a broad diffraction peak centred at approximately 38°, which correspond to the results reported in [40, 41], indicating an amorphous structure. The high mixing entropy and large difference in atom size clearly increased the complexity in the as-deposited HEA coating, which can promote the formation of an amorphous structure. The XRD patterns of the AlCrTaTiZr-based nitride and oxide coatings showed only diffraction peaks of the substrate, which indicates that the nitrides and oxides were amorphous structures.

Figure 4 shows the current-density profiles as a function of the permeation-testing time for the as-deposited HEA coatings. The electrochemical hydrogen permeation test of each specimen was repeated three times. With increasing test time, all samples reached a steady-state. The steady-state hydrogen permeation current density of the as-deposited AlCrTaTiZr HEA coating was approximately $1.83 \times 10^{-6}$ A cm$^{-2}$, and the difference between the steady-state hydrogen permeation current density and the beginning of hydrogen charging was approximately $7.50 \times 10^{-7}$ A cm$^{-2}$. The steady-state hydrogen permeation current density of the as-deposited
AlCrTaTiZr HEA nitride coating was about approximately 1.78 × 10^{-5} A cm^{-2}, and the difference between the steady-state hydrogen permeation current density and the beginning of hydrogen charging was about approximately 9.10 × 10^{-6} A cm^{-2}. In addition, the steady-state hydrogen permeation current density of the as-deposited AlCrTaTiZr HEA oxide coating was approximately 1.79 × 10^{-5} A cm^{-2}, and the difference between the steady-state hydrogen permeation current density and the beginning of hydrogen charging was about approximately 1.69 × 10^{-5} A cm^{-2}. All these electrochemical hydrogen permeation results indicate that the HEA oxide coating and nitride coating had nearly the same hydrogen permeation resistance. In this case, the HEA coating may have better hydrogen permeation resistance than the other two samples.

Generally, only a few micrometres of metallic layer on a substrate does not confer better hydrogen permeation resistance compared with oxides or nitrides [42]. Surface oxidation or nitridation results in a lower permeability, while in our present study, both oxidized HEA and nitrided HEA coatings showed greater hydrogen permeability. This phenomenon most likely occurred because oxides or nitrides were precipitated inside the boundaries and stress fields could have enhanced permeation. In addition, based on the obtained results of SEM surface and cross-sectional morphology analysis, the HEA coating was more compact than the oxide and nitride coatings, as shown in figures 2(a)–(c).

A coating with a dense and compact surface morphology and a more effective hydrogen isotope permeation resistance can be obtained. In addition, comparing the cross-sectional morphologies of these coatings, it was found that the HEA coating had the densest cross-sectional morphology, while the nitride and oxide coatings were obviously consisted coarse columnar crystals. Notably, the coarse columnar crystals of the nitride and oxide coatings penetrated the entire coating and directly reached the substrate, which was completely different than the cross-sectional morphology of the HEA coating, as shown in figures 2(d)–(f). Generally, the grain boundary forms a channel for the rapid diffusion of deuterium, and the columnar crystal directly reaching the substrate allows deuterium to reach the surface of the substrate along the grain boundary [37, 39]. Therefore, columnar crystals penetrating the entire coating directly to the substrate significantly reduce the deuterium permeation resistance of coatings [43]. Furthermore, the increase of the electrochemical hydrogen permeation suggests either the reduction of oxide or nitride, or hydridation of Ta, Ti and Zr. Since HEA oxide and nitride coatings include the hydriding elements of Ta [44], Ti [45] and Zr [46], they can convert to hydrides during the electrochemical hydrogen permeation process, which would further enhance their permeation. This was highly likely in this study because the gas permeation did not change the permeation, as shown in figure 5.

The results of electrochemical hydrogen permeation can provide a valuable reference, which is crucial to optimize the coating preparation process. However, the test results of the gas-driven deuterium permeation test of the tritium permeation barriers (TPBs) obtained at high temperature are indispensable for evaluating the deuterium permeation resistance of coatings. Figure 5 presents the gas-driven deuterium permeation curves of AlCrTaTiZr-based coatings. As the figure shows, the deuterium ion current was recorded during the heating process from 550 °C to 700 °C, and during the cooling process from 700 °C to 550 °C. All the tested coatings with a thickness of approximately 1.2 μm were prepared for the deuterium permeation test according to the obtained deposition rate as indicated by the SEM analysis, as shown in figure 2. In this test, the HEA nitride coating exhibited a much lower deuterium permeation flux than the other two samples, which indicates that the HEA nitride coating had better deuterium permeation resistance than the other two samples. However, the HEA

Figure 4. Electrochemical hydrogen permeation curves of the AlCrTaTiZr-based coatings.
oxide coating had the highest permeation flux in this temperature range, which indicates that the HEA oxide coating had the worst deuterium permeation tolerance. Generally, surface oxidation or nitridation results in better deuterium permeation resistance, but in our current study, the HEA coating had a better deuterium permeation resistance than the HEA oxide coating. In most cases, for a reduction in deuterium permeation, the surface oxide is critically important. In this study, all constituent elements of the HEA were very likely oxidized by residual H2O in a D2 atmosphere. In particular, the oxidation of Al, Ta and Zr could not be avoided. If selective oxidation occurred, the HEA coating could have a better deuterium permeation resistance than the HEA oxide coating. The microstructural changes of the coatings after annealing treatment will be analysed in the following sections.

Nearly the same deuterium permeation flux was found between the heating process and the cooling process during the gas-driven deuterium permeation experiment, which indicates that the HEA oxide coatings may have good thermal stability. In addition, the HEA coatings presented an obvious difference in deuterium permeation flux during the heating and cooling processes. The structure of the HEA coating may have changed when the heating temperature reached 700 °C, and thus, the deuterium permeation flux changed during the heating and cooling processes. Furthermore, it was clearly shown that the deuterium permeation flux of the nitride coating is significantly different during the heating and cooling processes. This phenomenon could have been the result of changes in the microstructure of the HEA nitride coating with the increase of the deuterium permeation test temperature, which would make the deuterium permeation resistance of the coatings significantly better after the test temperature reached 700 °C.

The temperature dependence of the permeability for the HEA coatings is shown in figure 6. All deuterium permeabilities were obtained at a deuterium driving pressure of \(7 \times 10^4\) Pa. As shown in the figure, the HEA oxide coating had the worst deuterium permeation resistance, and the permeability was nearly same during the heating and cooling processes. The HEA nitride coating had the best deuterium permeation resistance. The permeability of the HEA and nitride coatings during the cooling process was significantly lower than that during the heating process, which could be related to the structural change of the coatings at high temperatures.

### 3.2. Microstructure and hydrogen permeation resistance after annealing treatment

Figure 7 presents the surface morphology of the HEA-based coatings after annealing treatment. Figure 7(a) shows the surface morphology of the HEA coating after annealing treatment, which was completely different from that of the surface morphology before annealing treatment, as shown in figure 2. Obvious fine particles appeared on the surface of the HEA coating, without obvious holes or cracks, as shown in figure 7(a). The corresponding elemental composition of the HEA coating after annealing treatment is shown in figure 7(b). As shown in the figure, a large amount of O was evident in the coating, which indicates that the surface of the HEA coating was oxidized after annealing treatment. Figure 7(c) shows the surface morphology of the HEA nitride coating after annealing treatment. A large number of scratches appeared on the surface of the coating, which...
were caused by incomplete polishing of the surface of the substrate. In addition, there was no obvious change in the surface of the coating after annealing treatment. A small number of large particles adhered to the coating surface, which could indicate that the microstructure of the HEA nitride coating changed after annealing treatment.

Figure 7(d) presents the surface morphology of the HEA oxide coating after the annealing treatment. As shown in the figure, the surface of the coating became loose, and a large number of pores appeared on the surface of the coating after annealing treatment. Notably, the HEA coating was very likely oxidized by residual H2O in deuterium gas-driven permeation test. The oxidized HEA coating had a compact surface morphology compared
to the HEA oxide coating, which could have been the main reasons that the HEA coating showed better deuterium permeation resistance than the HEA oxide coating. In addition, a loose surface morphology and pores appeared in the HEA oxide coating, which could have been the main reasons that the HEA oxide coating had the worst deuterium permeation resistance.

To further verify the evolution of the microstructure of the coatings during the gas-driven deuterium permeation test process, XRD analysis was performed. Figure 8 presents the XRD patterns of the AlCrTaTiZr-based HEA coatings after annealing treatment. There were several newly formed diffraction peaks that appeared in the HEA nitride coating compared with the XRD pattern before annealing treatment, as shown in figure 3. All these newly formed diffraction peaks were BCC crystal structures, without the formation of other single metal oxides. Generally, the constituent AlN, CrN, TaN, TiN and ZrN nitrides have a face-centred cubic (FCC) crystal structure [47]. The changed deuterium permeation flux of the HEA nitride coating during the cooling process, shown in figures 5 and 6, could have been related to such a change in microstructure. In addition, a weak diffraction peak centred at approximately 29° appeared on the HEA coating, which indicates that the microstructure of the HEA coating changed after annealing treatment, which is consistent with the analysis of the surface morphology of the HEA coating shown in figures 7(a) and (b). The change in the deuterium permeation flux shown in figures 5 and 6 during the heating and cooling processes, may be consistent with the microstructure change that occurred during the permeation experiment.

Notably, in the XRD patterns shown in figure 8, no obvious diffraction peak appeared in the HEA oxide coating after annealing treatment. The absence of a new diffraction peak for the HEA oxide coating after annealing treatment, indicates that the phase structure of the oxide coating did not change significantly and had relatively good phase stability. This could be the main reason that the permeation flux of the HEA oxide coating showed little change during the heating and cooling processes, as shown in figures 5 and 6.

Figure 9 presents electrochemical hydrogen permeation curves of AlCrTaTiZr-based coatings after annealing treatment. The steady-state electrochemical hydrogen permeation current density of the HEA oxide coatings was approximately $8.09 \times 10^{-6}$ A cm$^{-2}$, and the difference between the steady-state current density and the initial hydrogen charging current density was approximately $5.60 \times 10^{-6}$ A cm$^{-2}$. The HEA coatings had a steady-state current density of approximately $4.78 \times 10^{-6}$ A cm$^{-2}$, and the difference between it and the beginning of hydrogen charging was approximately $2.30 \times 10^{-6}$ A cm$^{-2}$. In addition, the steady-state current density of HEA nitride coatings was approximately $4.01 \times 10^{-6}$ A cm$^{-2}$, and the difference between it and the beginning of hydrogen charging was about approximately $2.49 \times 10^{-6}$ A cm$^{-2}$. In this case, the HEA nitride coating and HEA coating had nearly the same hydrogen permeation resistance, and the HEA oxide coating had the worst hydrogen permeation resistance. All these electrochemical hydrogen permeation results of the AlCrTaTiZr-based HEA coatings after annealing treatment are completely opposite to those of the electrochemical hydrogen permeation results of the AlCrTaTiZr-based coatings before annealing treatment, as shown in figure 4. The annealed HEA coating had better hydrogen permeation resistance than the HEA oxide coating, which corresponds to the result of gas-driven deuterium permeation, as shown in figures 5 and 6. Combined with the results shown in figures 7–9, it can be concluded that the oxidation of the HEA coating...
during the deuterium permeation experiment and the change in the microstructure could be the main reasons that the HEA coating had a better deuterium permeation resistance and why a change in deuterium permeation flux during the heating and the cooling processes, as shown in figures 5 and 6.

4. Conclusion

In this work, AlCrTaTiZr, (AlCrTaTiZr)N and (AlCrTaTiZr)O HEA coatings were prepared by magnetron co-sputtering. The crystalline structures of the coatings were studied by XRD and confirmed to be amorphous structure. The results of electrochemical hydrogen permeation revealed that the AlCrTaTiZr coating had the best hydrogen permeation resistance, and the (AlCrTaTiZr)N and (AlCrTaTiZr)O coatings had nearly the same hydrogen permeation resistance. However, the D-GDP experiments showed that the (AlCrTaTiZr)N coating had the best deuterium permeation resistance, and that the (AlCrTaTiZr)O coating had the worst deuterium permeation resistance. Annealing experiments were performed to investigate the change in the microstructure of the coatings during D-GDP experiments, and the results indicated that the change in the coating microstructure was the main reasons for the difference in the deuterium permeation flux between the heating and cooling processes. In addition, the oxidization of the HEA coating during the deuterium gas-driven permeation process was the main reasons that the HEA coating has a better deuterium permeation resistance than the HEA oxide coating. Selecting a better high entropy alloy system and annealing treatment process will help to improve the properties of deuterium permeation resistance.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

ORCID iDs

Wei Zhang https://orcid.org/0000-0002-8470-400X
Xiaofang Luo https://orcid.org/0000-0002-1172-1992
Xiaoyu Wang https://orcid.org/0000-0002-7302-8560
Jijun Yang https://orcid.org/0000-0002-1945-3312
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