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ARTICLE

Microstructure and Diffusion Mechanism of CoCrCuFeNi HEA Joints Diffusion Welded Using Co Filler

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Abstract: A CoCrCuFeNi high-entropy alloy (HEA) was diffusion welded using a Co filler at 850, 950, 1050, and 1100 °C, and the microstructure and diffusion mechanism of the joints were examined. Results show that firm connections are achieved at each temperature, no intermetallic compound forms in the joints, and some Kirkendall voids are remained on the HEA side around the interface. The diffusion coefficients of Cr, Fe, Ni, and Cu in the Co filler at 850 and 950 °C are calculated, ranked as follows: Cu>Cr>Fe>Ni. The diffusion rates of all elements are at the same level. The diffusion between the CoCrCuFeNi HEA and Co filler occurs under the combined action of the vacancy and grain boundary diffusion mechanisms.

Key words: high-entropy alloy; diffusion welding; microstructure; diffusion coefficient; diffusion mechanism

High-entropy alloys (HEAs) are novel materials composed of five or more principal elements in equal or near-equal molar ratios[1]. Because of their four core effects, namely, high-entropy, severe lattice distortion, sluggish diffusion kinetics, and cocktail effects, HEAs have simple microstructures and excellent properties, such as high strength, high thermal stability, remarkable wear resistance, and good corrosion resistance[2–3]. They have broad applications in aerospace, nuclear equipment, weapons manufacturing, and other fields[4]. Therefore, the welding technology of HEAs should be studied to promote their industrial application.

Diffusion welding is a solid-phase joining method, which is advantageous for the welding of dissimilar materials, ceramics and intermetallics. The diffusion welding of an HEA to a dissimilar-material usually has poor compatibility and requires the insertion of a filler metal that has good compatibility with both the HEA and the other material. As HEAs are emerging materials, studies on their welding to dissimilar materials are insufficient, and the existing research mainly focuses on the joining of HEAs and superalloys, steels, Ti alloys, Al alloys, Cu, and a few nonmetals.

In investigations of the weldability between HEAs and superalloys, the Inconel 718 superalloy was welded to an Al0.1CoCrFeNi HEA by gas tungsten arc welding[5], brazed to an FeCoNiTiAl HEA with a BNi2 filler[6], and diffusion welded to a CoCrFeNiMo medium-entropy alloy using a Ni filler[7]. An AlCoCrFeNi2.1 eutectic HEA and GH4169 were diffusion welded by Li et al[8], an Inconel 718 was laser welded by Bridges et al[9] using a NiMnFeCoCu HEA filler, and a ZrB_2-SiC-C ceramic was brazed to GH99 by Zhang et al[10] using a Ti-modified FeCoNiCrCu HEA filler.

Studies on dissimilar-material welding between HEAs and steels are mainly about the welding of CoCrFeMnNi HEAs and stainless steels, such as 316[11], duplex[12], and 304[13]. A CoCrCuFeNi HEA and 304 stainless steel were diffusion welded by Li et al[14], and their tensile test shows that the joints are all broken in the base metal area.

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Some researchers have joined HEAs and aluminum alloys; a FeCoCrNiMn HEA and Al 1060 were friction stir lap welded by Yao et al[15], an AlCoCrFeNi HEA and Al 6061 were explosive welded by Arab et al[16], and an Al0.3CrFe1.5 MnNi0.5 HEA and Al 6061 were directly active-soldered in air with a Sn3.5Ag4Ti filler by Tsao et al[17].

Some studies have reported the welding of HEAs with copper or titanium; Wang et al[18] laser-welded Ti-6Al-4V and FeCoNiCrMn using a Cu filler, Ding et al[19] diffusion-welded copper to titanium using a CoCrFeMnNi HEA filler, and Liu et al[20] studied the interfacial behavior and joint performance of CoCrFeMnNi HEA/pure Cu joints achieved by vacuum diffusion welding.

A few studies on the joining of HEAs and nonmetals have been published; an Al0.85CoCrFeNi HEA and a TiAl intermetallic alloy were vacuum diffusion welded by Lei et al[21], and a SiC ceramic was brazed with a CoCrCuFeNi HEA filler by Wang et al[22].

HEAs are usually used as filler metals to join dissimilar materials; a CoNiCuNb0.5V1.5 HEA filler was used by Gu et al[23] to laser-weld TC4 and Al 6082, CoCrFeNiCu HEAs were used as filler metals by Hao et al[24] to join TC4 and 304 stainless steel, a AlCoCrFeNi HEA filler was used by Azhari-Saray et al[25] to weld Al 6061 to St-12 carbon steel, and a CoCrFeMnNi HEA filler was used by Ding et al[19] to diffusion weld copper to titanium.

Studies on the welding of CoCrCuFeNi HEAs are scarce despite their mature preparation process and excellent comprehensive performance. Moreover, the research on weldability between HEAs and dissimilar materials is insufficient. The performance of joints between CoCrFeNi-based HEAs and titanium alloys is usually deteriorated by the brittle intermetallic compounds Ti-Fe and Ti-Cr, and Cu as a filler metal can reduce brittle compounds[18,24,26]. However, Cu segregates in the intergranular owing to its small bonding energy with Fe, Co, Ni, and Cr, resulting in a high tendency of hot cracking[27–29]. $Al_{13}Fe_4$- and Al_5Fe_2-type intermetallic compounds form in the joints of FeCoCrNiMn HEAs and Al 1060 despite the use of a low heat input during friction stir welding[15]. CoCrFeNi-based HEAs have good weldability with nickel and its alloys[5,7–8] and stainless steels[11–14].

With the development of HEA preparation and processing, the dissimilar-material welding between HEAs and other materials, such as metals, ceramics, and intermetallics, will increase. A filler metal is needed in case of poor weldability between HEA and other materials. Nevertheless, the research on the weldability and bonding characteristics of HEAs with other metals is insufficient.

With good heat and corrosion resistance, cobalt and its alloys can be used as matrices to prepare superalloys[30]. Here, we hypothesize that cobalt and CoCrCuFeNi HEAs do not react to form intermetallic compounds, and this is demonstrated by the experiments in this work. Cobalt has considerable potential as a suitable filler metal for welding HEAs to some dissimilar materials. However, to the best of our knowledge, neither the weldability between CoCrCuFeNi HEAs and Co nor the practicability of welding CoCrCuFeNi HEAs using Co fillers has been studied. In this work, a CoCrCuFeNi HEA was diffusion-welded using a Co filler at different temperatures. The microstructure and diffusion mechanism of the joints were discussed in detail to provide theoretical support for the selection of filler metals in the diffusion welding of CoCrCuFeNi HEAs to other materials. The results are also significant for the dissimilar-material diffusion welding of CoCrCuFeNi HEAs and cobalt-based materials.

1 Experiment

CoCrCuFeNi ingots were fabricated using a vacuum induction furnace and cast in a graphite mold (200 mm×100 mm× 3 mm). The ingots were all melted at least five times to ensure their homogeneity, and they were cut into dimensions of 10 mm×10 mm×3 mm and 15 mm×10 mm×3 mm via electric spark cutting. The raw materials were pure Co, Cr, Cu, Fe, and Ni powders with a purity of 99.9%. The CoCrCuFeNi HEA was composed of an fcc solid solution (intragranular) and a small Cu-rich phase (intergranular), as shown in Fig.1a; the compositions of the two phases are displayed in Fig. 1b and consistent with existing reports[9,14,29,31]. During the solidification of the CoCrCuFeNi HEA ingots, due to the influence of Cu segregation, the Cu content of the HEA intragranular is low (about 8.84at%), whereas that of the intergranular is high (73.07at%). The filler metal was a commercial Co filler with a purity of 99.99% and a thickness of 30 μm.

Before welding, the workpieces were polished using abrasive paper and ultrasonically cleaned using alcohol. A

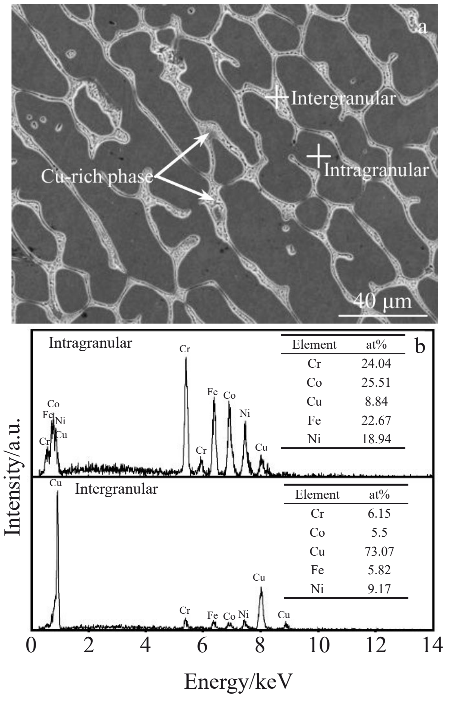

Fig.1 Microstructure (a) and EDS results of intergranular and intragranular (b) of CoCrCuFeNi

schematic of the assemblies of workpieces to be welded is shown in Fig. 2. The diffusion welding parameters are shown in Table 1. Microstructural characterization was performed by scanning electron microscopy (SEM; NovaSEM 450) with a backscattered electron (BSE) detector. The compositions of the joints were examined via electron probe X-ray microanalysis (EPMA; JXA-8530F PLUS), and the phase compositions were analyzed through X-ray diffraction (XRD; SmartLab 9).

2 Results

2.1 Microstructure and composition analyses of joints

Fig.3 shows the BSE interfacial morphology of the joints at different temperatures. All joints were effectively connected. The interfaces have no compound layers or discrete compounds. Some pores are distributed on the CoCrCuFeNi HEA side around the interface. The interface of the joint welded at 850 °C is clear and straight, and the pores are gathered at the interface (Fig. 3a). At the diffusion welding temperatures of 950 and 1050 °C, the interface is gradually blurred and bent, as shown in Fig. 3b and 3c, respectively. At 1100 °C, the interface is increasingly blurred and curved, and the weld seam is significantly narrowed (Fig.3d).

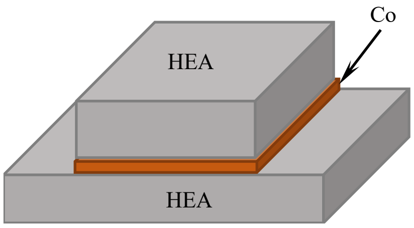

Fig.2 Schematic of assemblies

Table 1 Diffusion welding parameters

No.	Temperature/ °C	Interlayer	Holding time/ min	Pressure /MPa
1	850	Co	120	30
2	950			
3	1050			
4	1100			

The XRD patterns of the CoCrCuFeNi HEA base metal and the joints welded at different temperatures are shown in Fig.4. The HEA base metal and all joints contain fcc and Cu phases, which is consistent with previous reports[14,29]. All joints contain Co phases, whose diffraction peaks are consistent with that of Co powder in Ref. [32]. XRD analysis shows that no intermetallic compound forms in the welded joints.

The EPMA surface scanning test results of the joints welded at different temperatures are shown in Fig. 5. Each joint forms a diffusion layer at the interface between the CoCrCuFeNi HEA and Co filler, in which Co, Cr, Fe, and Ni are uniformly distributed, that is, without any element segregation. The distribution of Cu in the diffusion area is not clearly expressed in the surface scanning patterns because Cu is segregated in intergranular regions with atomic fractions exceeding 70at% in the HEA base metal, whereas the atomic fraction in the diffusion area is smaller than 5at%; consequently, the Cu in the diffusion layer is almost black and cannot be clearly distinguished. The distribution of Cu in the joints is discussed in the following EPMA line scanning analysis.

The diffusion layers of the joints welded at different temperatures were quantitatively analyzed by EPMA point

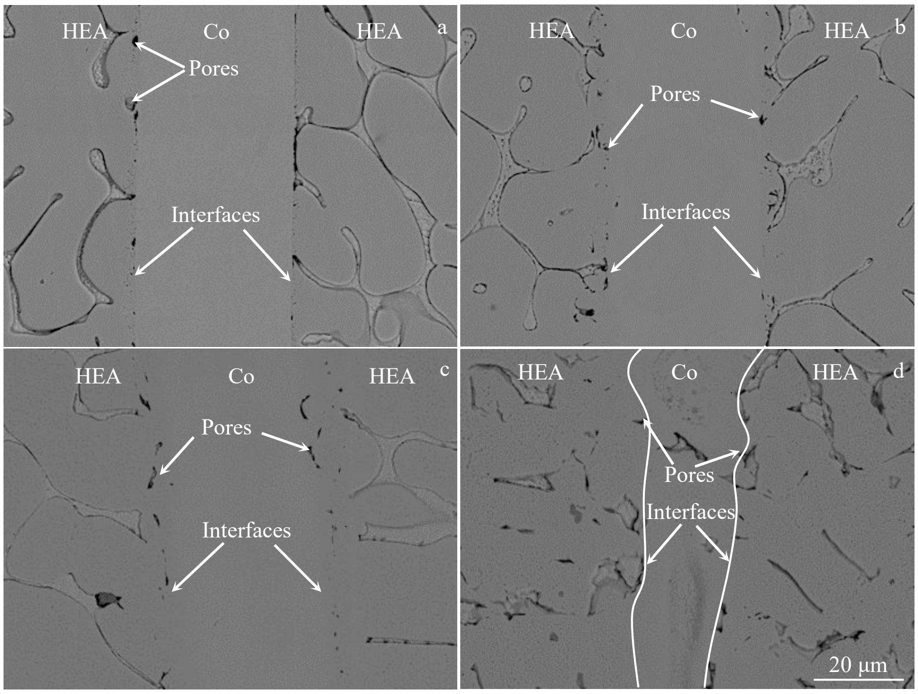

Fig.3 Interfacial morphologies of joints diffusion welded at different temperatures: (a) 850 °C, (b) 950 °C, (c) 1050 °C, and (d) 1100 °C

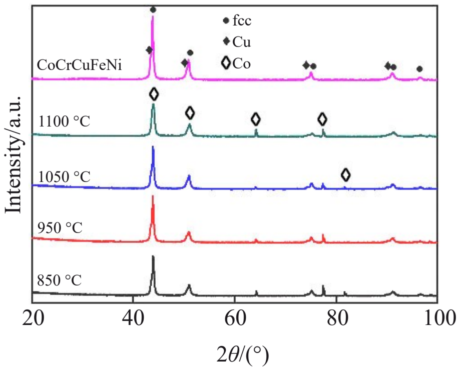

Fig.4 XRD patterns of CoCrCuFeNi alloy and joints achieved at different temperatures

scanning, and the results are shown in Table 2. Combined with the interface morphology, XRD analysis, and surface scanning results, the EPMA point scanning findings indicate that each diffusion layer is a Co solid solution rich in Cr, Fe, Ni, and Cu, and no intermetallic compounds form in the joints welded at different temperatures. The formation of intermetallic compounds often causes low ductility and brittle fracture of joints, which degrade joint performance[10,19].

At the welding temperature of 850 °C, the diffusion layer is thin and the interface is straight (Fig. 5a). At 950 °C, the diffusion layer is slightly thickened and the interface has a zigzag shape (Fig. 5b). As seen in Fig. 5a and 5b, the Cr, Fe, and Ni atoms are synchronously diffused from the CoCrCuFeNi HEA toward the Co filler at certain positions in the interfacial areas at 850 and 950 °C. At 1050 °C, Cr, Fe, and Ni still diffuse from the HEA base metal to the Co filler synchronously and Ni penetrates the Co filler in local areas (Fig. 5c). At 1100 °C, the penetration diffusion area of Ni increases and the penetration diffusion of Fe appears in local areas (Fig.5d).

EPMA line scanning was conducted on the joints welded at different temperatures to further analyze the element diffusion, and the results are shown in Fig. 6. The line scanning path in the HEA passes through the intragranular area, which has a low Cu content, but not through the intergranular area which has Cu segregation. Therefore, the content of Cu is lower than that of other elements in the line scanning results. No compositional ladder forms between the CoCrCuFeNi HEA and Co filler, which also proves the lack of intermetallic compounds at the interface. As seen in Fig. 6a and 6b, Cr, Fe, Ni, and Cu do not penetrate the Co filler. Thus, the diffusion process between the CoCrCuFeNi HEA and Co filler can be calculated using the diffusion couple model in Fick's second law, in which the compositions at both ends of the diffusion couples are not affected by diffusion. The diffusion coefficient of each element is calculated according to this law, as seen in the next section.

As shown in Fig. 6c and 6d, nearly all elements diffuse throughout the Co filler at 1050 and 1100 °C, so the abovementioned diffusion couple is no longer applicable. In particular, the diffusion of Cu is fast. At 1100 °C, the concentration of Cu in the weld seam is equal to that in the intragranular of CoCrCuFeNi HEA. Cr, Fe, and Ni have similar concentration distributions.

Combined with the surface scanning results, the line scanning findings indicate the following results. First, the Cu atoms diffuse into the Co filler faster than the Cr, Fe, and Ni atoms at 1050 and 1100 °C. Second, the diffusion rates of Cr, Fe, and Ni are close in most areas at 1050 and 1100 °C. Third, the Ni atoms more easily penetrate the Co filler in local areas at 1050 or 1100 °C. Fourth, the Cr, Fe, and Ni atoms have the same diffusion path at all diffusion temperatures.

In order to discuss the properties of the joints welded at different temperatures, shear tests were carried out. All shear tests were unloaded manually when they were unable to continue after the joints suffer severe plastic deformation. The bearing capacity of the joint welded at 850 ℃ reaches 481 MPa when the test is unloaded manually. It can be seen that the diffusion welded joints of CoCrCuFeNi HEA with Co filler have good shear strength and plastic deformation ability.

2.2 Calculation of diffusion coefficients

The diffusion coefficients of each element at 850 and 950 °C were calculated using the diffusion couple model in Fick's second law, where the compositions at both ends of the diffusion couples are not affected by diffusion. The relationship between the mass concentration of each element and the diffusion distance and time is described by the following error function:

$$\omega_{(x,t)} = \frac{\omega_1 + \omega_2}{2} + \frac{\omega_1 - \omega_2}{2}\operatorname{erf}\left(\frac{x}{2\sqrt{Dt}}\right) \tag{1}$$

where $\omega_{(x,t)}$ is the element mass concentration for diffusion time t and diffusion position x; ω_1 is the element mass concentration in the Co filler; ω_2 is the element mass concentration in the CoCrCuFeNi HEA base metal, which is the average values of 20 points in the intragranular; D is the diffusion coefficient of atoms; t is the diffusion time (7200 s in this study); and x is the position of the composition test point, that is, the distance from the composition test point to the original interface. The element concentration of the original interface is $(\omega_1+\omega_2)/2$.

Initial condition of equation (t=0):

$$\omega_{(x,0)} = \begin{cases} \omega_1, x < 0 \\ \omega_2, x > 0 \end{cases} \tag{2}$$

Boundary conditions ($t > 0$):

$$\omega_{(x,t)} = \begin{cases} \omega_1, x = -\infty \\ \omega_2, x = \infty \end{cases} \tag{3}$$

The intermediate variable is set to $\beta = \frac{x}{2\sqrt{Dt}}$. The value of β can be found according to the error function table.

$$D = \frac{x^2}{4\beta^2 t} \tag{4}$$

The diffusion coefficients of the Co atoms in the CoCrCuFeNi HEA at 850 and 950 °C are shown in Table 3, and those of the Cr, Fe, Ni, and Cu atoms in the Co filler are shown in Table 4. The calculated diffusion coefficients are at the same level with those in Ref. [19]. The diffusion

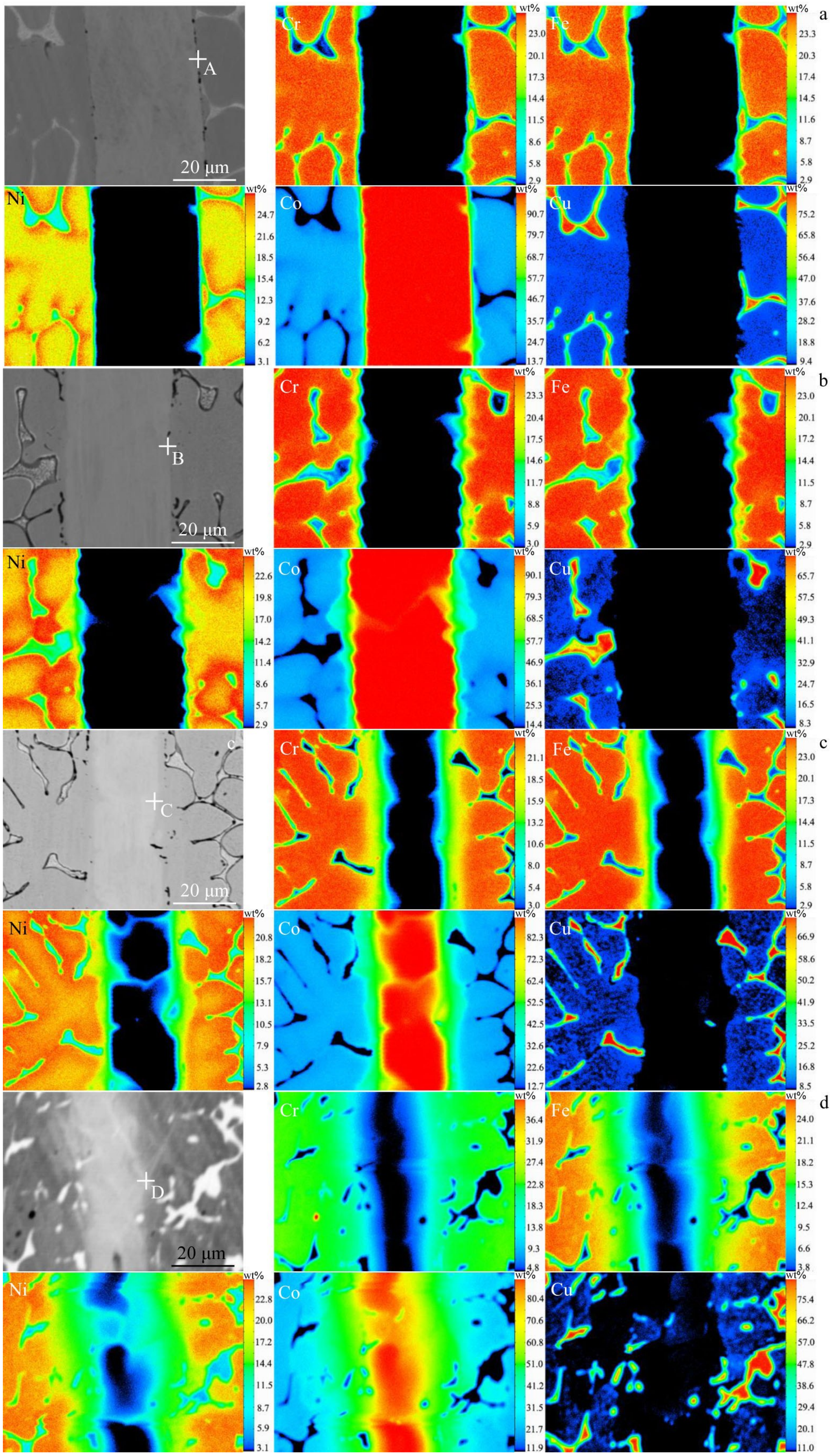

Fig.5 Morphologies and corresponding EPMA surface scanning results of joints achieved at different temperatures: (a) 850 °C, (b) 950 °C, (c) 1050 °C, and (d) 1100 °C

Table 2 EPMA results of points A–D in Fig.4 (wt%)

Point	Co	Cr	Fe	Ni	Cu
A	94.676	2.372	1.607	0.949	0.395
B	73.872	8.276	7.086	7.015	3.751
C	74.478	9.252	7.784	4.812	3.675
D	67.114	8.962	8.153	7.647	8.124

coefficients of all elements increase as the diffusion temperature improves from 850 °C to 950 °C. The diffusion rate of Co from the filler metal to the HEA base metal is consistently far smaller than the summed diffusion rates of Cr, Fe, Ni, and Cu from the HEA to the Co filler. Thus, Kirkendall voids form on the CoCrCuFeNi HEA side around the interface after diffusion welding (Fig. 3a and 3b). The diffusion coefficients of Cr, Fe, Ni, and Cu from the HEA to the Co filler at 850 and 950 °C are ranked as follows: Cu > Cr > Fe > Ni. At 850 °C, the diffusion coefficients of all elements are similar. At 950 °C, the diffusion coefficient of Co, Cr, Fe, and Cu is the same, whereas that of Ni is slightly smaller than that of other elements.

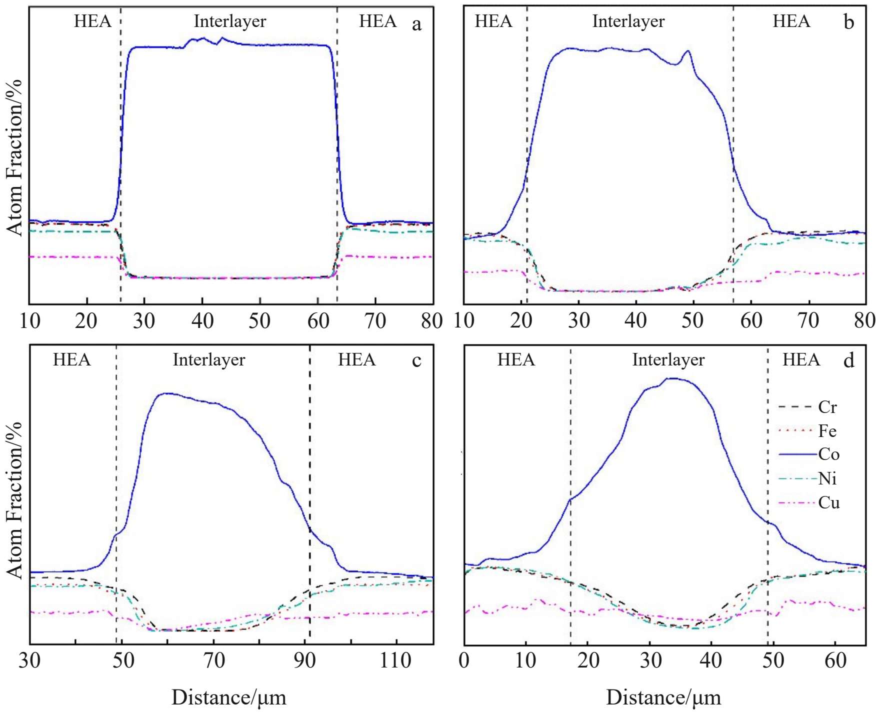

Fig.6 EPMA line scanning of joints welded at different temperatures: (a) 850 °C, (b) 950 °C, (c) 1050 °C, and (d) 1100 °C

The atom concentration distributions near the interface were calculated and then compared with the measured element distributions in Fig.7. The calculated and measured results fit well.

Table 3 Diffusion coefficients of Co in the CoCrCuFeNi HEA at 850 and 950 °C

Temperature/°C	850	950
Diffusion coefficient/×10^{-16} $m^2 \cdot s^{-1}$	0.3181	3.3115

Table 4 Diffusion coefficients of Cr, Fe, Ni, and Cu in the Co interlayer at 850 and 950 °C (×10^{-16} $m^2 \cdot s^{-1}$)

Temperature/°C	Co	Cr	Fe	Cu
850	0.4450	0.3591	0.3052	0.4726
950	3.8580	3.6131	0.6173	4.1605

3 Discussion

The experimental and calculation results suggest that the diffusion rate of Cu is slightly higher than that of other elements at all diffusion temperatures. The reason may be that Cu is more active than other elements due to its lower melting point. The melting points of Co, Cr, Cu, Fe, and Ni are listed in Table 5.

The diffusion rates of Ni atoms from the CoCrCuFeNi HEA to the Co filler are slower than that of Cr and Fe at 850 and 950 °C, which is consistent with the calculation results for the Cu/CoCrFeMnNi diffusion system of Ding et al[19]. Therefore, the diffusion resistance of Ni is relatively great at 850–950 °C. However, at 1050–1100 °C, the long-distance diffusion ability of the Ni atoms in the local area is significantly enhanced. The Ni atoms precede the Fe and Cr atoms in penetrating the weld seam (Fig. 5c and 5d). In general, the Cr, Fe, and Ni atoms have similar diffusion coefficients and paths from the HEA to the filler metal in most areas.

The diffusion between the CoCrCuFeNi HEA and the Co filler occurs under the combined action of the vacancy and grain boundary diffusion mechanisms. Atoms diffuse into a

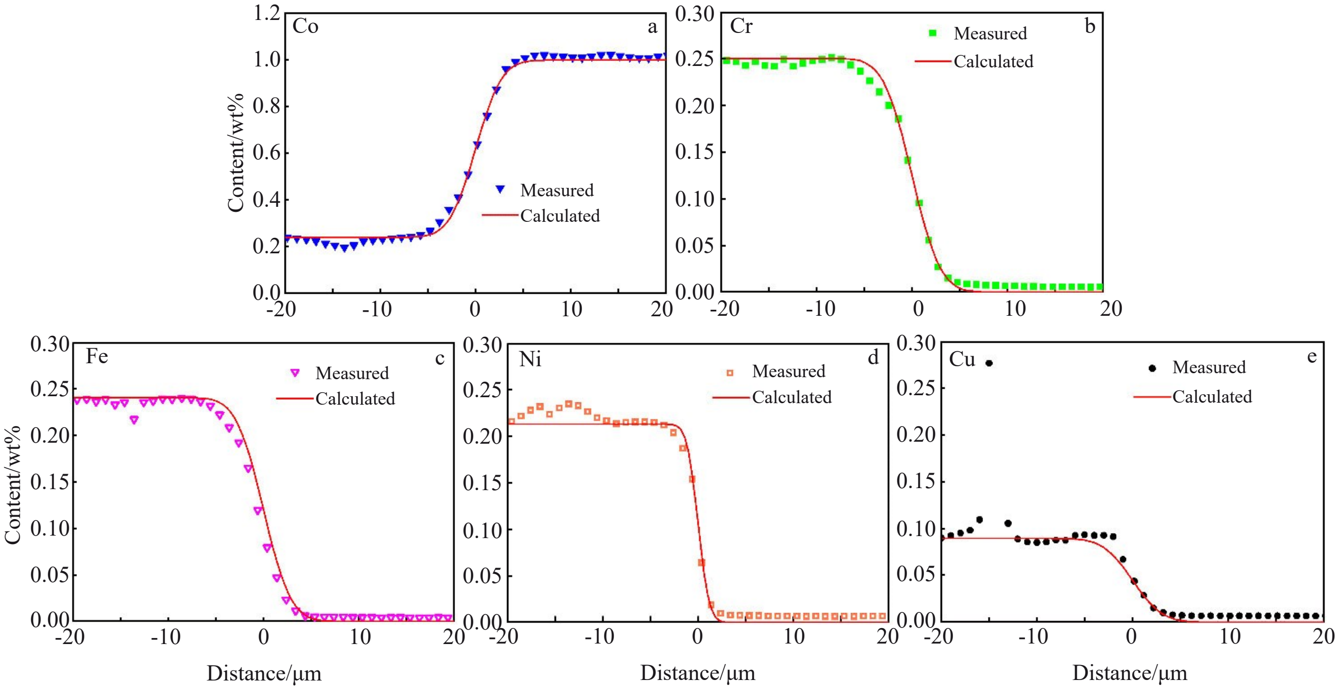

Fig.7 Measured and calculated composition distribution of each element produced at 950 °C**:** (a) Co, (b) Cr, (c) Fe, (d) Ni, and (e) Cu

Table 5 Melting points of Co, Cr, Cu, Fe, and Ni

Element	Co	Cr	Cu	Fe	Ni
Melting point/°C	1495	1907	1083	1538	1453

crystal through the vacancy mechanism. The higher the diffusion temperature, the greater the vacancy concentration in the crystal and the easier the atom migration. This is also the main reason for the increase in the diffusion coefficient of each element with temperature increasing from 850 ° C to 950 ° C. At grain boundaries and dislocation regions, the lattice distortion caused by crystal defects is conducive to atom migration, so the atom diffusion rate is higher than that inside the intact crystal. Atoms easily penetrate weld seams with long distances in these areas. This is why diffusion precursors form in the local areas, as seen in the EPMA surface scanning results (Fig. 5), and why all elements have the same long-distance diffusion path.

4 Conclusions

1) Firm connections of CoCrCuFeNi HEAs diffusion welded using a Co filler are obtained at each temperature, no intermetallic compounds form in the joints, and some Kirkendall voids are remained on the HEA side around the interface.

2) The diffusion coefficients of each element at 850 and 950 °C are calculated. The diffusion coefficients of Cr, Fe, Ni, and Cu in the Co filler are ranked as follows: Cu > Cr > Fe > Ni. The Cu atoms diffuse faster than other elements at all temperatures. The Ni atoms diffuse slower at low temperatures, but their long-distance diffusion ability is stronger than other elements at high temperatures. The diffusion rates of all elements are at the same level.

3) The diffusion between the CoCrCuFeNi HEA and Co filler occurs under the combined action of the vacancy and grain boundary diffusion mechanisms.

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填充Co的CoCrCuFeNi高熵合金扩散焊接头的组织和扩散机制

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摘 要：在850，950，1050和1100 ℃下，填充Co中间层对CoCrCuFeNi高熵合金（HEA）进行了扩散焊接，并对接头微观组织和扩散机制进行了分析。结果表明，在各温度下接头均形成了牢固的结合，接头无金属间化合物生成，高熵合金侧界面周围残留部分柯肯达尔孔。对Cr、Fe、Cu和Ni在Co填充层中的扩散系数进行了计算，排序如下：Cu>Cr>Fe>Ni。所有元素的扩散速度均在相同水平，CoCrCuFeNi高熵合金和Co填充层之间的扩散是在空位机制和晶界扩散机制的共同作用下发生的。

关键词：高熵合金；扩散焊；显微组织；扩散系数；扩散机制

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