Effects of Fe, Cr and Mn on inter-diffusion in CoNi-containing multiple principal alloys

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ABSTRACT

Inter-diffusion in eight alloys from the binary CoNi alloy to the quinary FeCrMnCoNi high entropy alloy (HEA) was studied by the diffusion couple experiments. It was found that the FeCrMnCoNi HEA does not exhibit sluggish diffusion in both the absolute temperature scale and the homologous temperature scale. Effects of specific elements were analyzed: Mn increases inter-diffusion in the absolute temperature scale but decreases it in the homologous temperature scale; Cr decreases inter-diffusion in both temperature scales; Fe decrease inter-diffusion in the absolute temperature scale but in the homologous temperature scale, the interaction between Fe and other elements could become important. The relationship between the pre-exponential factor and homologous activation energy indicates that the atomic diffusion process may not change qualitatively from binary to multiple principal alloys.

1. Introduction

High entropy alloys (HEAs), which are the systems with five or more principal elements in equi-atomic or nearly equi-atomic proportions [1], have attracted considerable attention over the last decade due to their outstanding properties, such as exceptional high-temperature mechanical properties [2,3], creep strength [4–7] and oxidation resistance [8–10]. Their excellent properties have been proposed to correlate with the four 'core effects', i.e., the high entropy, severe lattice distortion, sluggish diffusion and cocktail effects [11]. Among them, the sluggish diffusion effect, i.e., atomic diffusion is retarded in HEAs compared with the pure metals and traditional alloys, has been considered to result in their exceptional high-temperature properties [2,3] and the formation of nano-structures [12–14], and thus has been studied intensively [15–24].

The measurements of inter-diffusion in the FeCrMnCoNi HEA by Tsai et al. [15] show for the first time the sluggish diffusion effect, and this is attributed to the higher normalized activation energies (Q/T_m with Q the activation energy and T_m the melting temperature) of HEAs. Later, Miracle and Senkow [16] re-plotted the results in the absolute temperature scale and argued that the diffusivity of HEA was not lower than other conventional materials. Diffusion in HEAs should not be assumed

sluggish with a mere increase of the number of elements. This was also drawn by the tracer diffusion experiments of Vaidya et al. [17] in the equi-atomic CoCrFeNi and CoCrFeMnNi alloys, in which the effect of Mn was also analyzed.

Jin et al. [18] estimated the inter-diffusion coefficients of seven diffusion couples (CoNi, FeNi, FeCoNi, CrCoNi, FeCrCoNi, FeCrMnCoNi and FeCrPdCoNi) and found that the extrapolated diffusivities at the melting temperature of medium entropy alloys and HEAs are lower than the pure metals and binary alloys, and their values strongly depend on their constituents. Even diffusion couples from binary alloys to quinary HEAs were measured, these diffusion couples were only part of the sub-systems and thus the roles of specific elements couldn't be shown completely. As pointed out by Guruvidyathri et al. [20], sluggish diffusion cannot be taken as a generalized statement without a systematic investigation of the effects of individual elements on diffusion in HEAs. In this sense, Dąbrowa et al. [19] carried out a comparative study among inter-diffusion in the FeCrMnCoNi, FeCrCoNi, CrMnCoNi, FeMnCoNi and AlFeCrCoNi HEAs and found that the sluggish behavior was correlated prominently with Mn.

Regarding that the previous studies on the roles of different elements were not comprehensive and focused primarily on Mn [17,19], a series

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Table 1
The compositions of the alloys in the diffusion couples.

Symbol	Nominal composition (at.%)	Experimental composition (at.%)
1	$Co_{40}Ni_{60}/Co_{60}Ni_{40}$	$Co_{39.2}Ni_{60.8}/Co_{58.7}Ni_{41.3}$
2	$Fe_{33.3}Co_{23.3}Ni_{43.3}/$ $Fe_{33.3}Co_{43.3}Ni_{23.3}$	$Fe_{35.5}Co_{22.9}Ni_{41.6}/Fe_{34.8}Co_{42.2}Ni_{23.0}$
3	$Cr_{33.3}Co_{23.3}Ni_{43.3}/$ $Cr_{33.3}Co_{43.3}Ni_{23.3}$	$Cr_{33.7}Co_{22.8}Ni_{43.5}/Cr_{35.0}Co_{41.4}Ni_{23.9}$
4	$Mn_{33.3}Co_{23.3}Ni_{43.3}/$ $Mn_{33.3}Co_{43.3}Ni_{23.3}$	$Mn_{36.3}Co_{21.7}Ni_{42.0}/Mn_{36.2}Co_{40.1}Ni_{23.7}$
5	$Fe_{25}Cr_{25}Co_{15}Ni_{35}/$ $Fe_{25}Cr_{25}Co_{35}Ni_{15}$	$Fe_{25.0}Cr_{26.0}Co_{14.7}Ni_{34.3}/$ $Fe_{24.4}Cr_{26.8}Co_{33.6}Ni_{15.2}$
6	$Cr_{25}Mn_{25}Co_{15}Ni_{35}/$ $Cr_{25}Mn_{25}Co_{35}Ni_{15}$	$Cr_{25.1}Mn_{26.0}Co_{14.1}Ni_{34.8}/$ $Cr_{25.4}Mn_{26.6}Co_{32.5}Ni_{15.5}$
7	$Fe_{25}Mn_{25}Co_{35}Ni_{15}/$ $Fe_{25}Mn_{25}Co_{15}Ni_{35}$	$Fe_{26.2}Mn_{25.8}Co_{33.8}Ni_{15.2}/$ $Fe_{26.1}Mn_{24.8}Co_{14.3}Ni_{33.8}$
8	$Fe_{20}Cr_{20}Mn_{20}Co_{10}Ni_{30}/$ $Fe_{20}Cr_{20}Mn_{20}Co_{30}Ni_{10}$	$Fe_{21.2}Cr_{20.4}Mn_{19.3}Co_{9.6}Ni_{29.5}/$ $Fe_{20.0}Cr_{21.2}Mn_{20.4}Co_{28.5}Ni_{9.9}$

of binary and pseudo-binary diffusion couples from the CoNi binary alloy to the FeCrMnCoNi quinary HEA were studied herein; their nominal and final compositions are given in Table 1. The main diffusion elements of these couples are Co and Ni, and the composition difference between them are set to be 20 at.% to obtain clearly the diffusion fields. The compositions of other elements are set to be equal and the values are dependent on the specific number of alloy components. Compared with the work of Jin et al. [18], all the sub-systems of FeCrMnCoNi HEA are included in the present CoNi-based diffusion couples and thus the effects of specific elements (e.g., Fe, Cr and Mn) on diffusion can be shown.

2. Experimental

The alloys of the diffusion couples were prepared by the following procedures. First, the raw materials (e.g., Co, Cr, Fe, Mn and Ni elements with a purity better than 99.9 wt.%) were melted by a vacuum arc melting furnace in a Ti-gettered and high-purity argon atmosphere. To ensure chemical homogeneity, electromagnetic stirring was adopted during the melting process and each ingot was re-melted for at least five times in the water-chilled copper crucible, held at the liquid state for about 3 min and flipped before each melting process. Then, the alloy buttons were homogenized at 1400 K for 24 h in a vacuum quartz tube.

After annealing, the buttons were cut into discs of 5 mm × 5 mm in cross-section and 3 mm in thickness. The discs were ground and polished by the standard metallurgical procedures to obtain smooth surfaces. The polished surfaces of corresponding discs were fixed by the molybdenum plate and stainless screw. Then, the diffusion couples were annealed for 72 h in vacuum quartz tubes at 1200 K, 1250 K, 1300 K and 1350 K, respectively. After diffusion annealing, the quartz tube containing the diffusion couples was immersed in water and broken to quench the diffusion process. The diffusion couples were then removed from the clamping device, cut perpendicular to the interfaces between the alloys, ground, and polished.

The composition gradients in the diffusion couples were characterized before and after annealing using the energy-dispersive X-ray spectroscopy mappings (FEI, Helios G4 CX). Fig. 1 shows one example of the FeCrMnCoNi diffusion couple. Before annealing, there are sharp changes of Co and Ni composition gradients at the boundary, whereas after annealing, these composition fields become diffusive, meaning that diffusion of Co and Ni across the boundary happens. For Fe, Cr and Mn, their diffusion fields seem homogeneous even after annealing, indicating that their diffusion across the boundary is insignificant.

The composition profiles of the diffusion couples after annealing were further measured using the Electron Probe Micro-analyzer (EPMA, JXA-8100). The results at 1250 K were shown in Fig. 2 for example and the composition profiles of the main diffusion elements were fitted by the Boltzmann function. Obvious composition gradients appear for the main diffusion elements of Co and Ni. The background elements in the diffusion couples without Mn (Fig. 2a, b, 2c and 2e) don't show obvious composition changes, whereas for the four other cases with Mn (Fig. 2d, f, 2g and 2h), the composition profiles of background elements show a sine-wave shape near the interface, indicating uphill diffusion happens during annealing. Because the diffusivity difference between Co and Ni is small and both of them belong to the slow diffusion elements, the appearance of uphill-diffusion could be contributed to the chemical potential gradients caused by the addition of Mn, which make the thermodynamic factor larger than unity [22].

3. Analytical method

The Sauer-Freise (S–F) method [25], which does not rely on a precise determination of the position of the Matano plane, is now widely adopted for binary and quasi-binary diffusion experiments [15,18,21]. Regarding that the inter-diffusion coefficients of the two main elements are not completely the same using directly the S–F method [25], a modified approach was proposed by Paul [26] to solve the problem, which is followed in the present study.

According to the Paul's approach [26], the compositions of an element can smoothened such that the sum of them is equal to one (i.e. $\sum_{i=1}^{n} x_i = 1$, where x_i is the composition of component) and the modified compositions of Co and Ni can be calculated by $M_{Co} = x_{Co} + 0.5\left(\sum x_i \begin{smallmatrix} i \neq Co \\ i \neq Ni \end{smallmatrix}\right)$ and $M_{Ni} = x_{Ni} + 0.5\left(\sum x_i \begin{smallmatrix} i \neq Co \\ i \neq Ni \end{smallmatrix}\right)$. Then the normalized modified compositions are $M_{Co(N)} = \frac{M_{Co}}{M_T}$ and $M_{Ni(N)} = \frac{M_{Ni}}{M_T}$ where $M_T = M_{Co} + M_{Ni}$. The inter-diffusion coefficient at a location is given by:

$$\widetilde{D}\left(Y^*_{M_{i(N)}}\right) = \frac{1}{2t(dY_{M_{i(N)}}/dx)_{x^*}}\left[(1-Y_{M_{i(N)}})\int_{-\infty}^{x^*} Y_{M_{i(N)}}dx + Y_{M_{i(N)}}\int_{x^*}^{+\infty}(1-Y_{M_{i(N)}})dx\right] \quad (1)$$

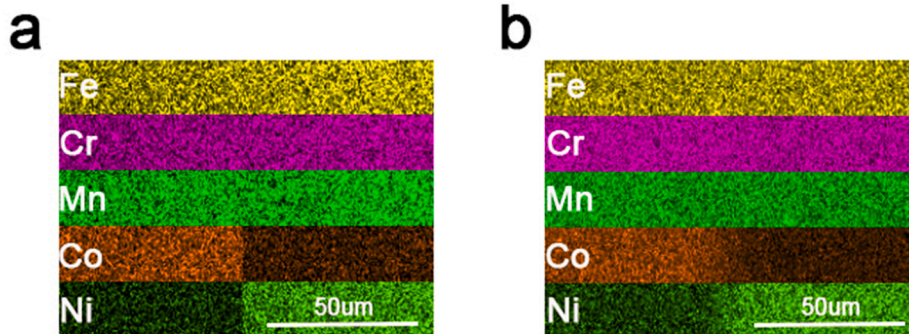where t is the annealing time and the normalized parameter $Y_{M_{i(N)}}$ is

Fig. 1. The energy-dispersive X-ray spectroscopy mappings of FeCrMnCoNi diffusion couple before (a) and after (b) annealing.
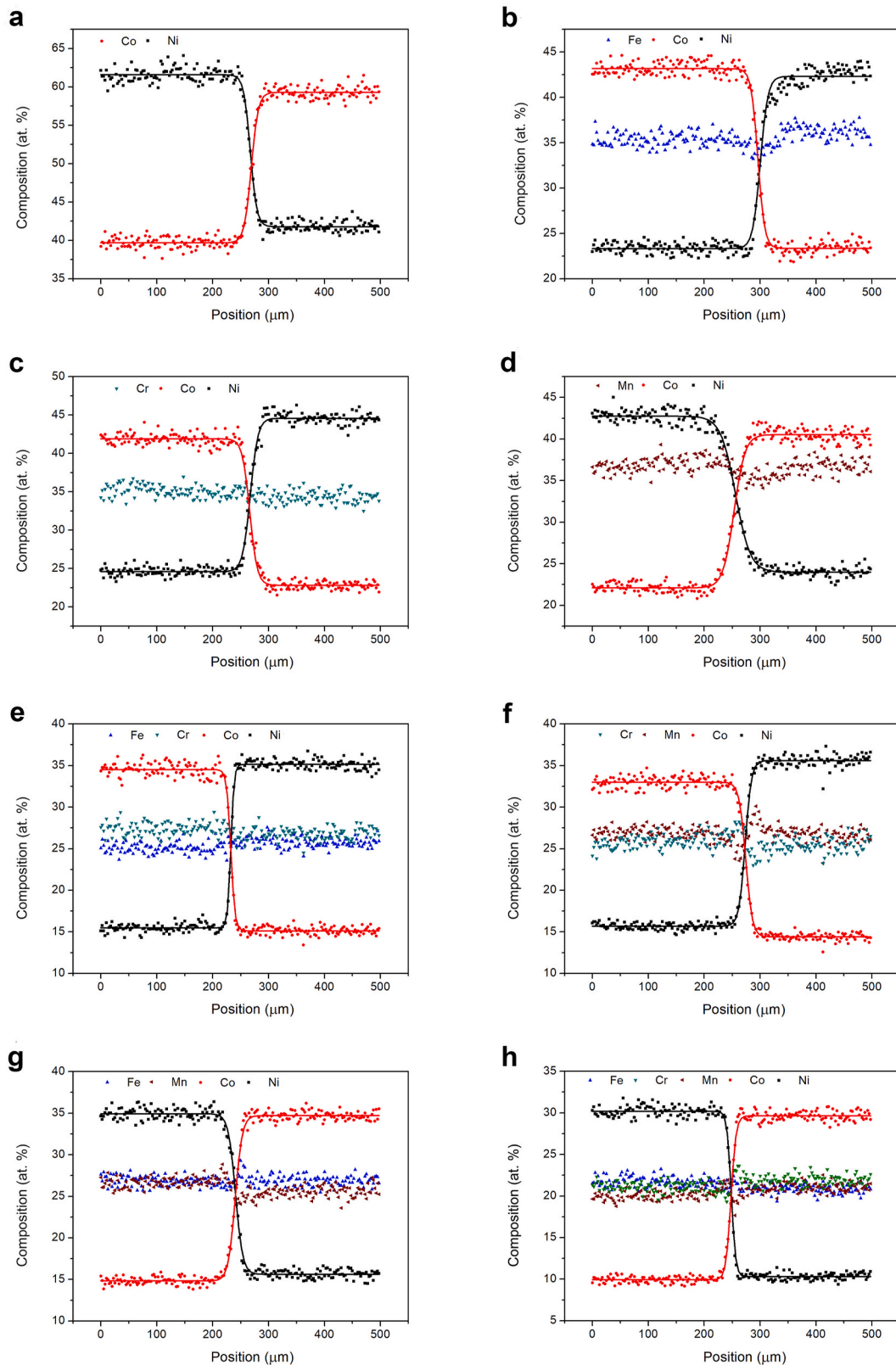
Fig. 2. Composition profiles of CoNi (a), FeCoNi (b), CrCoNi (c), MnCoNi (d), FeCrCoNi (e), CrMnCoNi (f), FeMnCoNi (g) and FeCrMnCoNi (h) diffusion couples annealed at 1250 K. The samples represent the experimental results while the lines are the fitting results of the Boltzmann function.

equal to $\frac{M_{i(N)} - M_{i(N)}^-}{M_{i(N)}^+ - M_{i(N)}^-}$.

The estimated concentration-dependent inter-diffusion coefficients obtained for the FeMnCoNi diffusion couple are representatively shown in Fig. 3, where the values close to both ends of diffusion couple are ignored due to the artifacts caused by numerical procedures. At the diffusion region, the diffusion coefficients don't exhibit obvious change with the increase of composition.
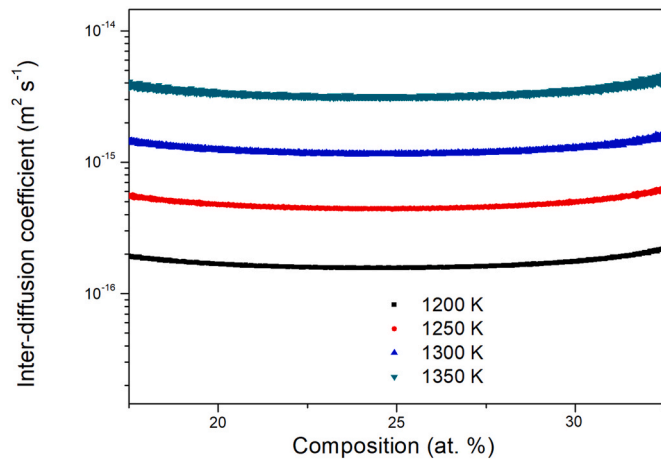
Fig. 3. Composition-dependent inter-diffusion coefficients of FeMnCoNi diffusion couple.

4. Results and discussion

Regarding that the inter-diffusion coefficients do not change obviously in the diffusion region, the inter-diffusion coefficient at the middle positions were chose for a further study. To compare the diffusivities in different alloys intuitively, the inter-diffusion coefficients were evaluated for the whole temperature range of current experiments and the results are shown in Fig. 4, where the logarithm of diffusion coefficient is plotted as a function of the absolute temperature and the homologous temperature. The melting temperatures for the alloys are from the work of Wu [27] as listed in Table 2.

In the case of absolute temperature (Fig. 4a), the inter-diffusion coefficient in FeCrMnCoNi alloy is not the smallest one, meaning that diffusivities will not always decrease with the increase number of alloy components, which was also mentioned in Jin's work [19]. After a comparison of the diffusivities between CoNi and MnCoNi, FeCoNi and FeMnCoNi, FeCrCoNi and FeCrMnCoNi, one can see that the addition of Mn increases the inter-diffusion coefficients obviously. In contrast, the addition of Cr decreases the inter-diffusion coefficients obviously, which can be found from comparisons of diffusivities between CoNi and CrCoNi, MnCoNi and CrMnCoNi, FeCoNi and FeCrCoNi, FeMnCoNi and FeCrMnCoNi. As for Fe, from CoNi to FeCoNi, from MnCoNi to FeMnCoNi, from CrCoNi to FeCrCoNi, and from CrMnCoNi to FeCrMnCoNi, one can see that its effect is similar to Cr.

In the case of homologous temperature as shown in Fig. 4b, like other literatures [17–19], the addition of Mn retards diffusion, which can be found by the changes of diffusion between CoNi and MnCoNi, CrCoNi and CrMnCoNi, FeCoNi and FeMnCoNi, FeCrCoNi and FeCrMnCoNi. The effect of Cr is the same as Mn but not as outstanding by comparisons between CoNi and CrCoNi, MnCoNi and CrMnCoNi, FeCoNi and

FeCrCoNi, FeMnCoNi and FeCrMnCoNi. The case of Fe is different. For CoNi and FeCoNi as well as CrCoNi and FeCrCoNi, the addition of Fe decreases the diffusivity while for MnCoNi and FeMnCoNi as well as CrMnCoNi and FeCrMnCoNi, the addition of Fe increases the diffusivity, which was also found for tracer diffusion of Cr and Co in the work of Dąbrowa [19], where the tracer diffusion coefficients were obtained by a optimization-based technique combining the Miedema's thermodynamic description with the diffusion couple experiments.

As has been mentioned, the effect of Mn on diffusion has attracted more attention than other elements [17,19]. According to Vaidya [17], the addition of Mn decreases the melting temperature of alloys, meaning that at a given temperature, such alloys have higher equilibrium concentration of vacancies, thus leading to enhanced substitutional diffusion. Though direct conclusions on the effects of Cr and Fe are few, there are some similar phenomena. The inter-diffusion coefficients in CrCoNi and FeCrCoNi are the smallest one in Jin's work [18], in which the effects of specific element cannot be shown completely because the diffusion couples are only part of the sub-systems. Further, recent work on dendrite growth in undercooled HEAs [28] shows a similar sluggish effect of Cr. The melting points of alloys don't change with the addition of Cr and Fe as shown in Table 2. The mixing enthalpies of Cr and Co as well as Fe and Co are negative, meaning that the atomic bonding between Co and other elements becomes stronger, which might retard inter-diffusion [17]. A similar behavior can be found for Ni as well.

The addition of Fe in MnCoNi and CrMnCoNi increases the inter-diffusion coefficient in homologous temperature, which implies that the sluggish effect is relatively independent of the number of elements [19] but is related highly to the specific elements. In this sense, one can see that not only the addition of Mn as found by Dąbrowa [19] but also the addition of Cr decrease diffusion in the homologous temperature scale. The effect of Fe is more complex as above analyzed and the addition of Fe to different alloys results in different effects, implying that the interaction between Fe and other elements might play a role.

The activation energy Q and the pre-exponential factor D_0 are determined by the Arrhenius equation:

$$D = D_0 \exp(-Q / RT) \tag{2}$$

where R is the gas constant. The obtained Q and D_0 are listed in Table 2, together with results from other literatures [17–19,21,30]. Fig. 5a shows the variation of D_0 in different alloys and Fig. 5b shows the variation of Q and homologous activation energy Q/T_m. one can see clearly that the alloy which has the higher value of activation energy also has the higher value of pre-exponential factor. So the reason for slower diffusion could not simply be attributed to the higher activation energy. For example, the FeCrMnCoNi HEA has the highest activation energy but its diffusion coefficient is not the smallest one. The variation tendency of Q/T_m is the same to that of Q even the melting temperature of alloys with Mn is much lower than that of alloys without Mn.

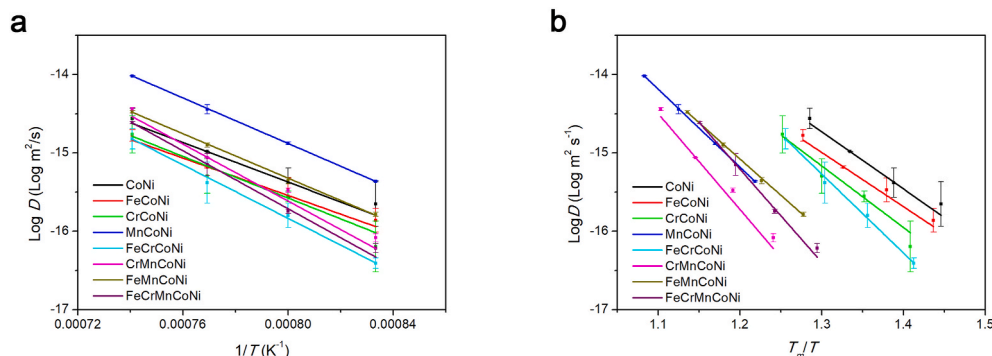According to the thermodynamic treatment by Dienes [29], D_0 can

Fig. 4. Arrhenius curves of inter-diffusion coefficients vs. the absolute temperature (a) and the homologous temperature (b).

Table 2
Melting temperatures, activation energy and pre-exponential factors for the alloys.

Alloys	Diffusivity	T_m (K)	Q (kJ mol⁻¹)	D_0 (m² s⁻¹)
CoNi	$\widetilde{D}_{CoNi}$	1735 [27]	242.105	5.56×10^{-6}
	$\widetilde{D}_{CoNi}$	1735 [27]	268 [18]	5.3×10^{-4} [18]
FeCoNi	$\widetilde{D}_{CoNi}$	1724 [27]	228.289	9.94×10^{-7}
	$\widetilde{D}_{CoNi}$	1724 [27]	286 [18]	1.6×10^{-4} [18]
CrCoNi	$\widetilde{D}_{CoNi}$	1690 [27]	254.437	1.14×10^{-5}
	$\widetilde{D}_{CoNi}$	1690 [27]	330 [18]	35×10^{-4} [18]
MnCoNi	$\widetilde{D}_{CoNi}$	1473 [27]	278.183	5.57×10^{-4}
FeCrCoNi	$\widetilde{D}_{CoNi}$	1695 [27]	326.725	6.58×10^{-3}
	D_{Co}	1717 [17]	240 [17]	4.8×10^{-7} [17]
	D_{Ni}	1717 [17]	253 [17]	1.1×10^{-6} [17]
	$\widetilde{D}_{CoNi}$	1695 [27]	309 [18]	4.9×10^{-4} [18]
	D_{Co}	1710.77 [19]	396.9 [19]	2.0×10^{0} [19]
	D_{Ni}	1710.77 [19]	374.8 [19]	7.98×10^{-2} [19]
	D_{Co}	–	247 [21]	1.6×10^{-2} [21]
	D_{Co}	–	299 [21]	6.3×10^{-1} [21]
	D_{Ni}	–	273 [21]	1.0×10^{-1} [21]
	D_{Ni}	–	306 [21]	1.0×10^{0} [21]
CrMnCoNi	$\widetilde{D}_{CoNi}$	1489 [27]	348.163	8.61×10^{-2}
	D_{Co}	1500.82 [19]	270.0 [19]	1.05×10^{-4} [19]
	D_{Ni}	1500.82 [19]	508.7 [19]	3.74×10^{4} [19]
FeMnCoNi	$\widetilde{D}_{CoNi}$	1533 [27]	271.787	1.08×10^{-4}
	D_{Co}	1543.49 [19]	314.5 [19]	4.18×10^{-3} [19]
	D_{Ni}	1543.49 [19]	243.5 [19]	2.8×10^{-6} [19]
FeCrMnCoNi	$\widetilde{D}_{CoNi}$	1553 [27]	354.188	1.22×10^{-1}
	D_{Co}	1607 [17]	270 [17]	1.6×10^{-5} [17]
	D_{Ni}	1607 [17]	304 [17]	6.2×10^{-4} [17]
	$\widetilde{D}_{CoNi}$	1553 [27]	308 [18]	9.8×10^{-4} [18]
	D_{Co}	1571.91 [19]	336.2 [19]	1.5×10^{-2} [19]
	D_{Ni}	1571.91 [19]	314.1 [19]	1.19×10^{-3} [19]
	D_{Co}	–	290.2 [30]	2.16×10^{-4} [30]
	D_{Ni}	–	265.0 [30]	1.47×10^{-5} [30]

Fig. 5. The pre-exponential factors (a), the activation energy and the normalized activation energy (b) of alloys in the current study. (c) The pre-exponential factor as a function of the normalized activation energy of alloys in the current and previous studies.

be correlated to Q and T_m by:

$$D_0 = \nu\lambda^2 K \exp(Q / RT_m) \tag{3}$$

where ν is the Debye frequency, λ is the lattice parameter and K is a constant. The correlation between the pre-exponential factor and the homologous activation energy is shown in Fig. 5, where the results from Refs. [17–19,21,30] are also included. Good linear fitting in Fig. 5c indicates that the relation in Eq. (3) is followed by both present and previous work. In other words, the atomic diffusion processes of Ni and Co may not be qualitatively changed with the increasing number of component as well as the addition of specific elements, as pointed out in Refs. [17,18].

5. Conclusion

In summary, inter-diffusion coefficients in the subsystems from binary alloy CoNi to FeCrMnCoNi HEA were estimated and analyzed. The following conclusion can be drawn.

(1) The sluggish diffusion effect does not exist in HEA for both cases of the absolute temperature and the homologous temperature scale;

(2) The specific elements could play a more important role than the number of elements in the diffusion process, e.g., Mn increases inter-diffusion in the absolute temperature scale but decreases it in the hom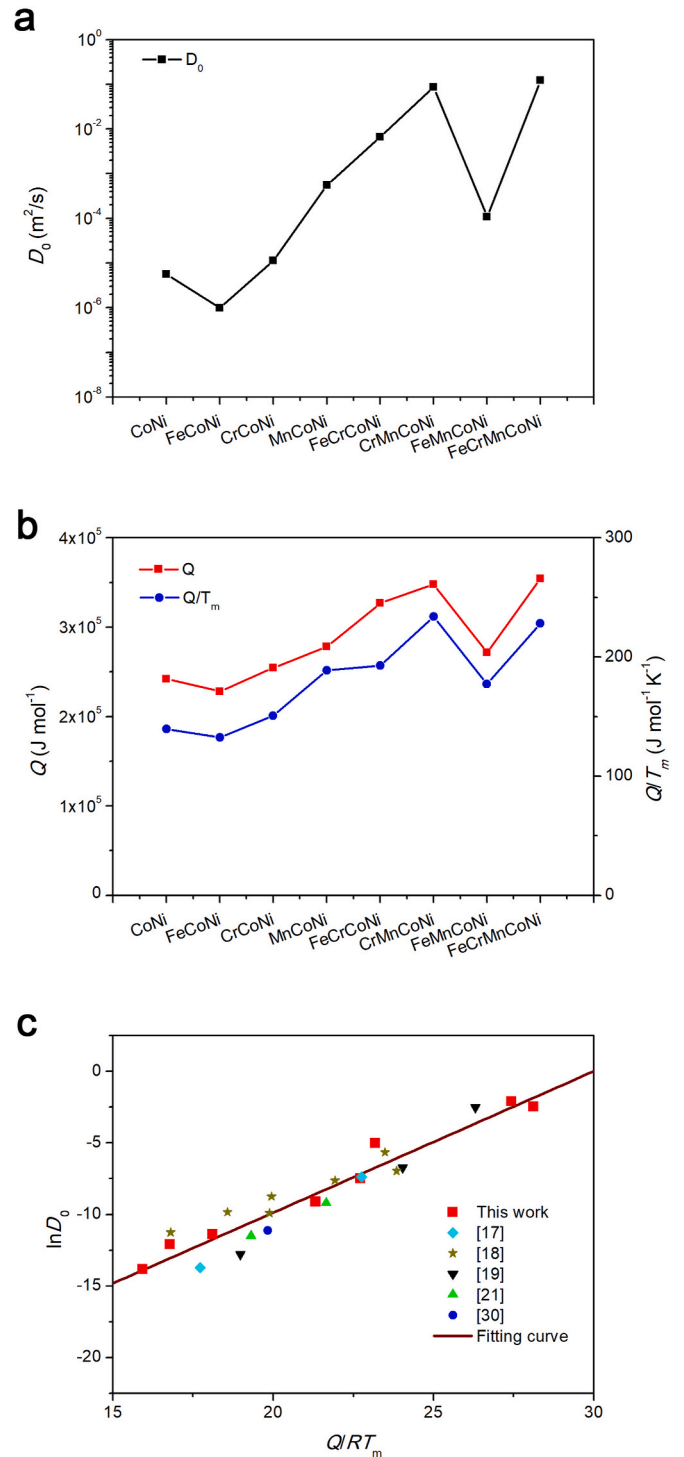ologous temperature scale, Cr decreases inter-diffusion in both temperature scales, and Fe decreases inter-diffusion in absolute temperature scale;

(3) The effect of interaction between different elements cannot be omitted, e.g., the interaction between Fe and other elements could influence the diffusion behavior in the homologous temperature scale;

(4) Both the number of components and the addition of specific elements don't qualitatively change the atomic diffusion process.

Credit author statement

Xin Li: Investigation, Validation, Visualization, Formal analysis, Software, Writing - Original draft preparation. **Jianbao Zhang*:** Conceptualization, Project administration, Investigation, Supervision, Writing - Reviewing & Editing. **Dexu Cui:** Investigation. **Xuan Luo:** Investigation. **Haifeng Wang**:** Resources, Writing - Reviewing & Editing, Supervision, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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