



Exceptional nanostructure stability and its origins in the CoCrNi-based precipitation-strengthened medium-entropy alloy

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recently, Zhao *et al.* reported that the coarsening rate of γ' nanoparticles in the $(\text{NiCoCrFe})_{94}\text{Al}_4\text{Ti}_2$ (at. %) HEA was much slower than that in the conventional Ni-based alloys [10]. They suggested that this remarkable thermal stability could be related to the sluggish diffusion of the HEA system. However, so far the intrinsic origins of this low coarsening rate are somewhat mysterious and reports on the early stages of phase separation are still missing, especially for the multi-component alloys involving complex interactions between various alloying elements.

As a result, in the present work, we have mainly focused on the detailed studies of the precipitation kinetics of the MEAs through combined experimental and computational analyses. The $(\text{CoCrNi})_{94}\text{Al}_3\text{Ti}_3$ (at. %) MEA, which exhibited a typical 'FCC + γ' ' dual-phase microstructure with excellent tensile properties [8], was chosen as a material for study here. The early-stage phase separation behavior and the coarsening kinetics of the γ' nanoparticles were carefully examined and discussed.

2. Experimental procedure

The $(\text{CoCrNi})_{94}\text{Al}_3\text{Ti}_3$ (at.%) MEA (referred as the Al3Ti3 MEA hereafter) was prepared by arc-melting the appropriate amounts of the pure elements under an Ar atmosphere. The resulting flats were then thermomechanically processed following the same procedure as reported in [8], including the solid-solution treatment (1200°C, 2 h), cold rolling ($\sim 66\%$) and recrystallization (1160°C, 3 min) leading to an equiaxed grain structure with $\sim 67\ \mu\text{m}$ grain size. Afterwards, additional long-term annealing treatments were performed at 800°C from 2 h to 360 h. Of particular interest, chemical

distributions of the as-recrystallized sample before aging treatment was detected to accurately determine the initial state of precipitation.

High-resolution XRD was carefully examined using a Rigaku X-ray diffraction (XRD) instrument by Cu-K α radiation with a monochromator, with a scan speed of 0.1 degrees/min. Lattice misfit can be derived by deconvolution of the (311) diffraction peak of alloy [11], following the equation of $\delta = 2(\alpha_{\text{particle}} - \alpha_{\text{matrix}}) / (\alpha_{\text{particle}} + \alpha_{\text{matrix}})$, in which α refers to the lattice parameter of each phase [12]. The microstructures of alloys were investigated by scanning electron microscopy (SEM, Quanta FEG450). Samples for SEM characterization were mechanically grounded and then electro-chemically polished in a solution of ethanol with the 4% nitric acid, at a voltage of 20 V. Atom probe tomography (APT) specimens were prepared using an FEI Scios dual-beam Microscopy instrument and the tip evaporation was performed using a CAMECA LEAP-5000XR device, with a 200 kHz pulse repetition rate, a pulse fraction of 20% at a set-point temperature of 70 K in the voltage-pulsing mode. Quantitative compositional analyses of the tips and associated 3D-reconstructions of atom maps were conducted by employing commercial software of IVAS.

3. Results and discussions

To start with, the early stage of precipitation, i.e. the as-solid-solution state, was carried out through APT (Figure 1). The frequency distribution analysis (FDA) in Figure 1(b) obtained from the APT data (Figure 1(a)) shows that all the elemental atoms closely follow the binomial distribution, with average Pearson coefficient for Co,

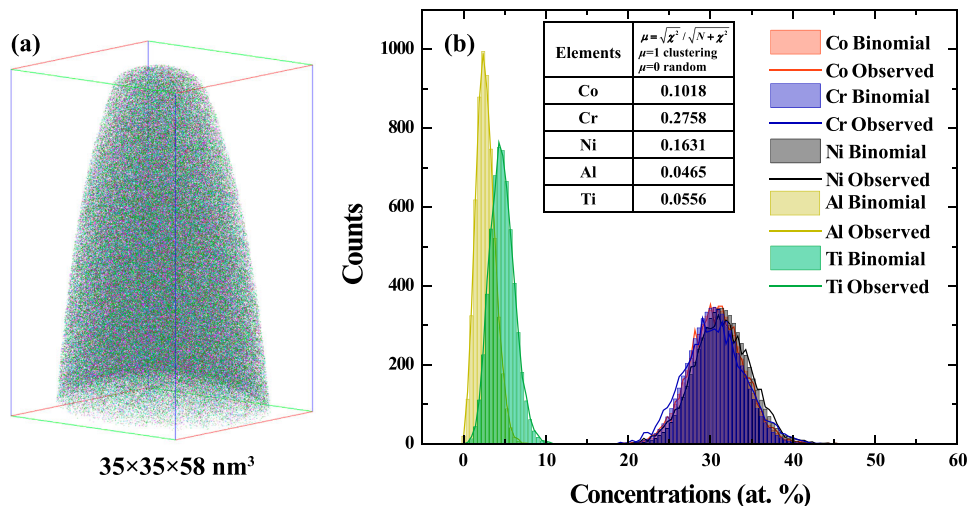


Figure 1. (a) APT map displays the atomic-scale elemental distribution. (b) Frequency distribution analysis for Co, Cr, Ni, Al and Ti atoms, which shows a comparison of observed (solid line) and the ideal random distribution (bar) described by binomial distributions for average solute contents.

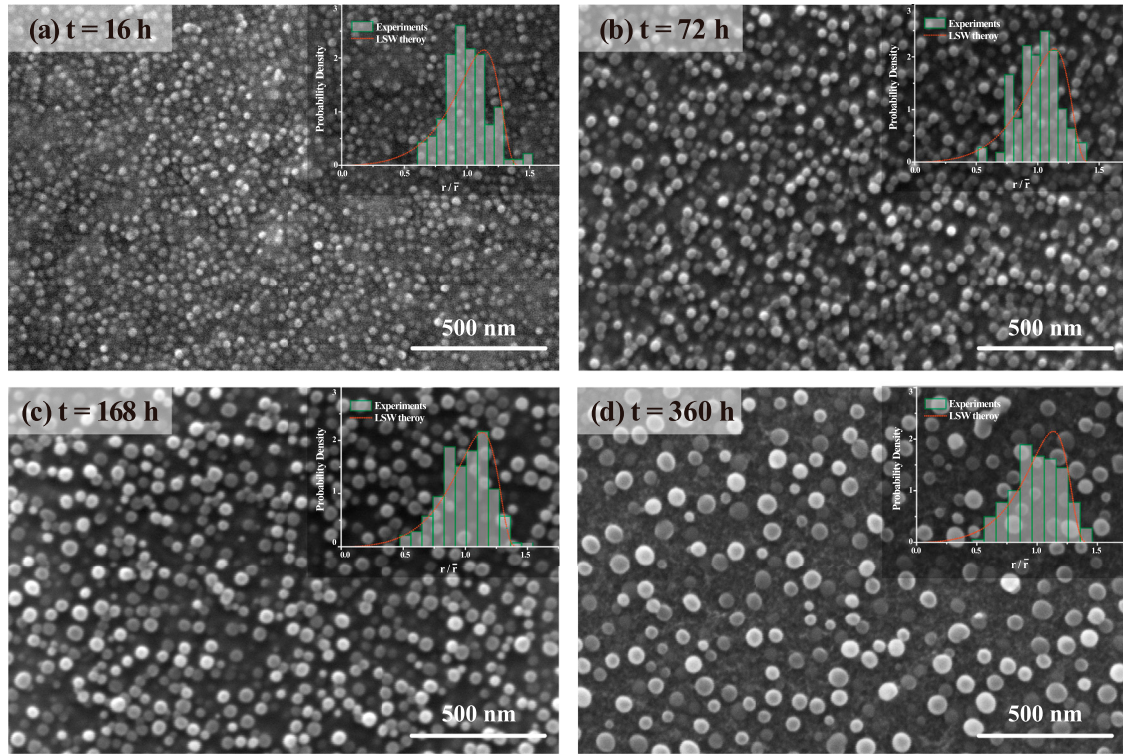


Figure 2. SEM images of the γ' precipitates in the Al₃Ti₃ alloy after aging at 800°C for (a) 16 h, (b) 72 h, (c) 168 h and (d) 360 h; The corresponding statistical particle size distribution of the experimental data and the prediction of the LSW model were also shown as insets.

Cr, Ni, Al and Ti of 0.1018, 0.2758, 0.1631, 0.0465, 0.0556, respectively, indicating a random distribution of individual elements in the sample without any formation of precipitates and clusters.

SEM images in Figure 2 show the particle distribution of the MEA aged at 800°C for various durations. The corresponding normalized particle size distributions (PSD) histograms analyzed by commercial ImageJ software are also presented accordingly. Three important parameters can be derived to quantify the microstructural evolution of the γ' nanoparticles in the Al₃Ti₃ MEA: (i) average particle size, (ii) particle size distributions (PSD), and (iii) growth rate of particles. Apparently, the average size of precipitated particles (in radius) increases as the aging time prolongs, ranging from 5.9 ± 0.6 nm after aging for 4 h to an average value of 25.7 ± 5.2 nm after aging for 360 h. The size distributions of the nanoparticles can be determined by measuring the fraction of nanoparticles of a given size that lies within the specific size intervals. The normalized probability density, $\rho^2 f(\rho)$, is determined using the following expression [13]

$$\rho^2 f(\rho) = \frac{N(r, r + \Delta r)}{\sum N_i(r, r + \Delta r)} \frac{\bar{r}}{\Delta r} \quad (1)$$

where $\bar{r}$ is the average radius of the precipitates and the normalized radius ρ is defined as the ratio of $r/\bar{r}$. For a clear comparison, the distribution functions predicted by the Lifshitz, Slyozov and Wagner (LSW) theory [14] are also superimposed here (red curves). Evidently, during the whole aging sequence, the size distribution of the γ' nanoparticles in the Al₃Ti₃ MEA shows a rather asymmetrical PSD with the peak values standing around $\rho = 1$, which is in reasonable agreement with the prediction by the LSW model. For the specimen aging at 360 h, a broad and flat PSD can be observed, implying that larger precipitates grow at the expense of the dissolution of the smaller ones.

It is also worth noting that unlike the conventional Ni-based superalloys, where the morphology of the γ' phase usually occurs in the sequence of spheres, cubes and ultimately solid-state dendrites as the coarsening is promoted by aging [12], the shape of the L₁₂ nanoparticles in our MEA is essentially maintained as spherical at all aging time. It is reported that a small lattice misfit between the matrix/precipitate interface would enable an increased critical size for the sphere-to-cube transition of the precipitates [15]. The lattice misfit of the present study was calculated from the deconvolution of XRD peak, to be $\sim 0.096\%$, which is well confirmed by our

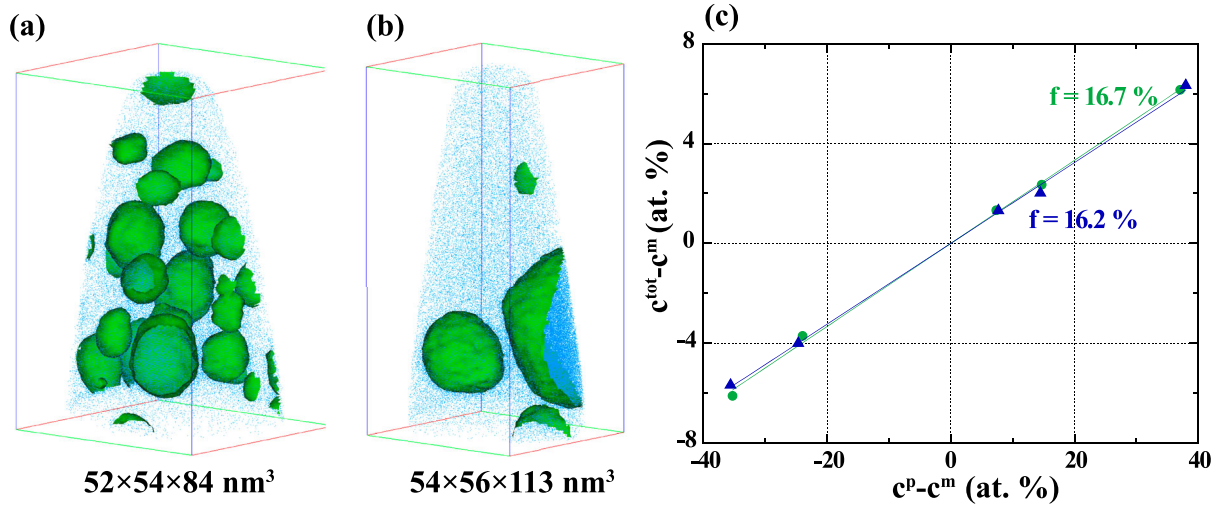


Figure 3. (a) and (b) APT-reconstructions of the Al₃Ti₃ MEA aged at 800°C for 4 h and 168 h, respectively; (c) Level rule for the calculation of the volume fraction of the γ' phase.

Table 1. Chemical compositions of γ' phase in the Al₃Ti₃ MEA (at. %) aged at 800°C for 4 h and 168 h.

	Phases	Compositions				
		Co	Cr	Ni	Al	Ti
4h	Matrix	34.46 ± 0.31	37.09 ± 0.31	25.80 ± 0.28	1.37 ± 0.08	1.27 ± 0.07
	Precipitate	10.53 ± 0.33	1.83 ± 0.14	62.92 ± 0.52	8.74 ± 0.31	15.99 ± 0.40
168h	Matrix	34.78 ± 0.28	37.30 ± 0.29	25.34 ± 0.26	1.30 ± 0.07	1.28 ± 0.07
	Precipitate	10.18 ± 0.31	1.73 ± 0.14	63.32 ± 0.50	9.00 ± 0.30	15.77 ± 0.38

observations. Similar morphology of the coherent γ' in the FCC matrix has also been reported in Ni-Cr-Al superalloy [16] and some other precipitation-hardened HEAs [10] with small values of lattice misfit.

The precipitation kinetics of the γ' nanoparticles in the present Al₃Ti₃ MEA were further analyzed in terms of growth rate. Figure 3(a,b) show 3D reconstructions related to the sample aging at 800°C for 4 and 168 h. For clarity, only the Ti element is displayed in the figures. The γ' nanoparticles are delineated by 10 at.% Ti iso-surfaces. Based on the elemental concentration obtained from the APT analysis (Table 1), the volume fractions of γ' nanoparticles were derived by the lever rule (as shown in Figure 3(c)), where c^p , c^m and c^{tot} are the concentrations of the individual elements in the nanoparticles, the matrix and the bulk material, respectively. It is evident that the volume fractions of γ' nanoparticles are kept almost constant at $\sim 16\%$ during the aging treatment from 4 h to 360 h. Thus, the microstructure evolution of the Al₃Ti₃ MEA within this stage could be approximately regarded as a stationary coarsening process (Ostwald ripening), which can be well described by the LSW theory [14], given as

$$r^n(t) - r^n(t_0) = k(t - t_0) \quad (2)$$

where k is the coarsening rate constant, $r(t_0)$ is the average size of nanoparticles at the aging time t_0 , referred as the time at the onset of coarsening. t is the time. n is the temporal exponent of the coarsening kinetics. If $n = 3$, the coarsening kinetic is governed by the volume-diffusion mechanism. For $n = 2$, it corresponds to the case that interface reaction is the rate-limiting step, related to the trans-interface diffusion controlled (TIDC) model [17]. To further determine the operative coarsening mechanism of our present case, the slope of a linear fit to the data points of $\log(r)$ vs. $\log(t)$ for isothermal aging at 800°C yields the inverse of the temporal exponent (about 0.3), suggesting a volume diffusion-controlled mechanism. Thus $n = 3$ is used to evaluate the temporal evolution of the particle size. Subsequently, the variation of r^3 as a function of time t for the γ' nanoparticles aged at 800°C is presented in Figure 4(a). Plots describing the coarsening behavior of the (FeCoNiCr)₉₄Al₂Ti₄ (at. %) are also inserted for a direct comparison [10]. According to the Equation (2), by linear fitting to the data set, the coarsening rate constant can be easily determined to be $k = 1.23 \times 10^{-29} \text{ m}^3/\text{s}$, which is obviously lower than its quaternary counterpart. More interestingly, such a slow coarsening rate of the γ' phase in the present Al₃Ti₃ MEA also remarkably outperforms most of the

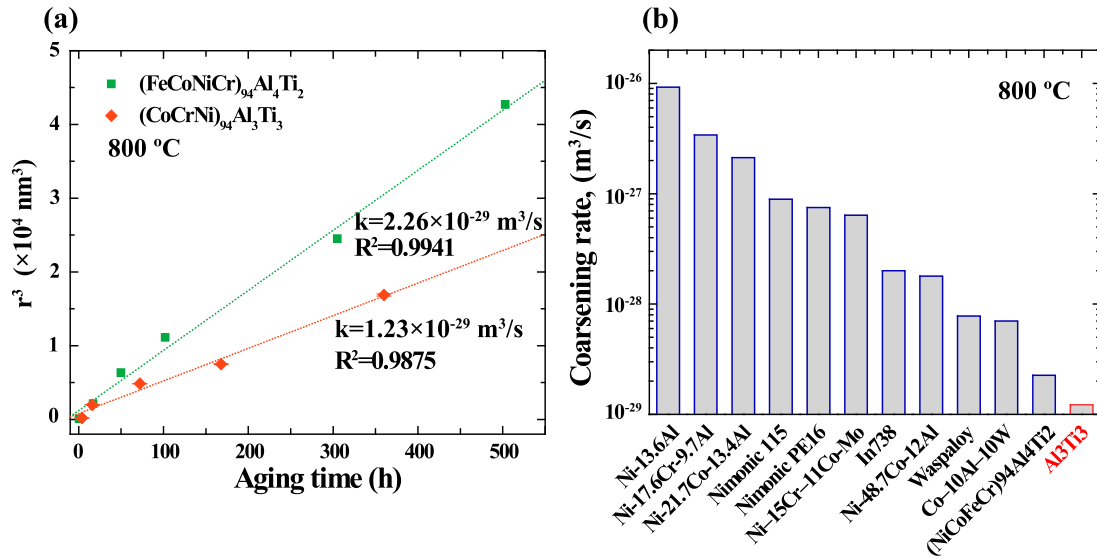


Figure 4. (a) The LSW relationship illustrating the coarsening of the γ' phase in the Al3Ti3 MEA and the reported (FeCoNiCr)₉₄Al₄Ti₂ MEA. (b) Comparison of coarsening rates for the Al3Ti3 MEA and a number of Ni-based superalloys at 800°C.

conventional γ' -strengthened superalloys (Figure 4(b)) [10,18,19,20–22].

The growth kinetics of the nanoparticles as well as its physical origins can be analyzed based on the ripening or coarsening theory. Theoretically, for the multi-component alloy systems, the coarsening rate constant k is a function of molar volume of the matrix (V_m), precipitate/matrix interfacial energy ($\sigma_{\gamma/\gamma'}$), the elemental mobility matrix (M) and the concentration gradient (Δc) of the diffusion species in the matrix and nanoparticles. For the original LSW model that is valid strictly for binary, ideal solutions, its applicability to multi-component alloys remains uncertainties. In a recent theoretical work, a more generalized treatment of coarsening in multi-component alloys accounting for off-diagonal terms in the diffusion tensor was proposed by Philippe and Voorhees (P-V) [23], where k can be expressed as:

$$k = \frac{8V_m\sigma_{\gamma/\gamma'}}{9(\Delta c)^T M^{-1} \Delta c} \quad (3)$$

Considering the thermal expansion from room temperature to 800°C, the value of V_m of the alloy is estimated using a rule-of-mixtures of the thermal expansion of the pure elements [10], approximated to be 7.12×10^{-6} m³·mol at 800°C. The appropriate atomic mobility parameters can be calculated using the NIST Ni-based mobility database [24] with ten elements involved, which have also been used to the diffusion evaluation in multi-component alloys [25]. The symmetric mobility matrix is then estimated based on the method proposed in references [24] and [26], which relate the mobility

Table 2. Mobility matrix for Al3Ti3 MEA calculated at 800°C for the γ -matrix composition determined via APT. Values are multiplied by 10⁻²³, and the units are m²·mol⁻¹·J⁻¹·s⁻¹.

k_i	Al	Ti	Co	Cr
Al	3.5407	-0.0830	-1.0502	-1.4679
Ti	-0.0830	3.7245	-1.1187	-1.5361
Co	-1.0502	-1.1187	12.6088	-7.1367
Cr	-1.4679	-1.5361	-7.1367	20.3425

matrix to the atomic mobility (M^*) of component i described in the Calphad mobility database. The calculated results were summarized in Table 2. Here Ni is chosen as the dependent composition variable. The value of $\sigma_{\gamma/\gamma'}$ can be easily obtained based on the Equation (3), given that the Δc has been accurately determined from the APT measurements (Table 1), yielding a remarkable small value of ~ 4 mJ·m² for 800°C. This value is located between the reported values from 3.7 to 8.1 mJ·m² for binary Ni-Al alloys [17,27]. Tiley *et al.* used 1.2 mJ·m² for their study of coarsening kinetics on the René 88 superalloys [28]. On account of the similar microstructure rendered by spherical coherent γ' nanoparticles embedded in the matrix, the interfacial energy we obtained here is quite acceptable. Such small interfacial energy certainly slows down the coarsening rate of the γ' nanoparticles, resulting in excellent thermal stability of this Al3Ti3 MEA.

In addition, the slow coarsening kinetics of the γ' nanoparticles in the Al3Ti3 MEA could also be correlated to the diffusion behavior. Actually, the hypothesis of the sluggish diffusion in multi-component alloy

systems is becoming a popular and controversial topic in more recent years [29,30], for which there is still not enough consensus on the validity and generality of this hypothesis [31,32]. Furthermore, so far no experiments have been conducted to evaluate the diffusion kinetics of our present case, and the chemical diffusion coefficient can only be calculated once accurate thermodynamic parameters are available. Fortunately, the diffusion behavior can also be preliminarily evaluated via the tracer diffusion coefficient, which is much easier to be obtained via the equation of $D = M^*RT$, where the atomic mobility of individual component, M^* , can be derived from the Calphad mobility database. T is the temperature, R is gas constant. As noted in the previous studies on various binary and ternary Ni-base superalloys, the diffusion of Al atom is generally regarded as the controlling step to determine the coarsening rate. This is primarily ascribed to the similar activation energy values ($260 \sim 280 \text{ J mol}^{-1} \cdot \text{K}^{-1}$) [28] for Al diffusion in the Ni-based superalloys and the Al diffusion in pure Ni. The tracer diffusivities for Ni, Co, Cr, Al and Ti were then estimated to be $\sim 7.5 \times 10^{-18}$, 2.8×10^{-18} , 9.0×10^{-18} , 2.5×10^{-17} , $2.6 \times 10^{-17} \text{ m}^2 \cdot \text{s}^{-1}$, respectively. Interestingly, based on the above calculations, we found that it is the Co atom that is the slowest diffusion component in the Al₃Ti₃ MEA, which is about one order of magnitude slower than that of the Al atom ($2.2 \times 10^{-17} \text{ m}^2 \cdot \text{s}^{-1}$) determined from the superalloys at the similar temperature [33]. In fact, Co did play a significant role in the coarsening behavior of the γ' particles in the Ni-based superalloys. With the increase of Co content, a reduction by a factor of 4 to 6 in γ' growth rate can be observed, which is probably due to the partitioning of cobalt between particle and matrix [21]. We should also indicate that great cautions should be taken for the values of those parameters, which could be varied with the reliability of the atomic mobility database and also the different precipitate coarsening models used to analyze the data. The arisen uncertainties for the derived interfacial free energies could be primarily due to the combined uncertainties in atomic mobilities and coarsening rate constant. For example, the uncertainties from the NIST Ni-based mobility database could be as high as 50% based on observed uncertainty in the diffusivity data [25,34], which may result in uncertainty with about the twice of the magnitude for the interfacial energy. Nevertheless, we can still safely conclude that it is either the small interfacial energy or the combined effect of slow diffusion behavior and the small interfacial energy that lead to the slow coarsening kinetics of the coherent γ' nanoparticles in the Al₃Ti₃ MEA. Certainly, additional researches for more accurate data especially the diffusion data are highly warranted for more precise analyses.

4. Conclusion

In summary, a remarkable sluggish coarsening behavior of the coherent γ' nanoparticles has been experimentally demonstrated in the (CoCrNi)₉₄Al₃Ti₃ (at. %) MEA, as compared to various conventional superalloys. The results indicated that such excellent thermal stability is mainly controlled by the volume diffusion mechanism. By using the model of Philippe and Voorhees in conjunction with the available atomic mobility data, it is surprisingly found that the interfacial energy for our present MEA is quite small with a value of $\sim 4 \text{ mJ} \cdot \text{m}^{-2}$. More importantly, unlike the traditional cases where the diffusion process is often dominated by the precipitates forming elements, such as the Al atom in the Ni-based alloy, the slowest diffusion species in the present MEA is found to be the Co atom. The diffusion coefficient was estimated in terms of the tracer diffusion coefficient, which is about one order of magnitude slower than that of conventional superalloys at the same temperature. Thus, it is accepted that this exceptional thermal stability of the L1₂-type nanoparticles could be ascribed to either the small interfacial energy or the combined effect of slow diffusion and small interfacial energy.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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