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Lightweight Al-Nb-Ti-V-Cr high entropy alloys with high hardness and enhanced mechanical properties via doping Co

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Abstract: To develop high-hardness and high-strength lightweight high entropy alloys (LHEAs), a series of Co_xAlNbTiVCr alloys were designed. The phase constitution, distribution, and crystal structure of the Laves phase in alloys can be altered by adjusting the composition of HEAs, which in turn influences their mechanical properties. Co_xAlNbTiVCr (*x*=0, 0.5, 1, 1.5, and 2, atomic ratio percentage) LHEAs were designed and prepared to characterize the microstructure and tailor the mechanical properties. The introduction of Co changes the microstructure of LHEAs from a single B2 structure to a mixture dendrite structure, which consists of B2 phase, C14 and C15 Laves phase. Wherein the C14 and C15 Laves phases exhibit coupled growth. Several parameters including mixing enthalpy (ΔH_{mix}), valence electron concentration (VEC), atomic radius size (δ), mixing entropy (ΔS), and electronegativity difference ($\Delta \chi$) are used to predict the formation of B2 and Laves phase in LHEAs. When the Co content increases from 0 to 1.5at.%, Laves phase volume fraction gradually increases, which leads to an enhancement in the compressive strength from 1,520.8 MPa to 1,844.4 MPa. Co_{1.5}AlNbTiVCr alloy exhibits the maximum Vickers hardness of 699.4 HV. The improvement of mechanical properties mainly originates from solid solution strengthening and second phase strengthening.

Keywords: lightweight high entropy alloy; Laves phase; microstructure; mechanical properties

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1 Introduction

Pursuit and expectation for materials with low density, good strength, and high ductility, have never faded in fields of energy conservation and environmental protection, engineering applications, and scientific research ^[1-3]. Efforts to reach the target applications of lightweight materials with excellent properties have been predominantly through alloying methods ^[4+12]. In recent years, in the quest for optimal mechanical

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properties among the seemingly countless choices of alloy compositions, a rapidly emerging paradigm, the so-called high entropy alloys (HEAs), exhibit significant advantages over traditional alloys ^[13-15]. Among them, AINbTiV lightweight high entropy alloy (LHEA) designed by Stepanov ^[16] has attracted widespread attention from materials researchers, which has a low density of 5.59 g·cm⁻³, but its strength is not high enough.

To further improve the mechanical properties of AlNbTiV LHEA, AlNbTiVCr_x LHEAs were prepared. The results demonstrate that introduction of Cr promotes the formation of the C14 Laves phase and enhances the room temperature mechanical properties of the alloys ^[17]. Similarly, Jiang et al. ^[18] prepared Al_{0.8}Nb_{0.5}Ti₂V₂Zr_{0.5} LHEA with C14 Laves phase by vacuum arc melting, and the alloys exhibit superior mechanical performance to nickel-based superalloy. Choi et al. ^[19] produced (CoCuFeNi)_{100-x}Zr_x LHEAs consisting of an FCC1, FCC2 matrix phase, and Laves phase. When *x* increased from 0 to 7, the compressive strength increased from 232 MPa to 446 MPa without decreasing its plasticity. Some researchers observed the transformation of the

C14 Laves phase into the C15 Laves phase during synchronized shearing. Yao et al. ^[20] found that the compressive yield strength of $Ti_3V_2NbNi_{0.5}$ alloy reinforced by the C15 Laves phase present in the interdendritic area reached 1,130 MPa with an ultimate compressive strain of 20%. Recent studies have conclusively verified that the atomic radius of Co is relatively small, and it can form C15 Laves phase with transition group elements with larger atomic radii (such as Zr and Hf)^[21,22].

In general, single-phase solid solution HEAs with equal mole ratios have limitations because of the trade-off relationship between different performance characteristics. To break through the limitations, intermetallic phases in multiphase alloys, especially Laves phases, become an important candidate to improve the performance of alloys ^[23-25]. Laves phase, which is the largest community of intermetallic compounds with many representatives, has the classic composition AB2 [26-28]. Laves phase exists in three types, which are the hexagonal MgZn₂ (C14), cubic MgCu₂ (C15), and hexagonal MgNi₂ (C36)^[29]. Numerous studies revealed that the formation criteria of Laves phase in HEAs are mainly determined by the difference in atomic radius sizes, the mixing enthalpy, and the valence electron concentration [30-33]. However, the synergistic interaction of multiple Laves phases and the formation of multiple Laves phases in HEAs are still poorly investigated. Hence, it is an attractive and challenging problem to investigate the strengthening mechanism of multiple Laves phases in HEAs.

In the present work, Co_xAlNbTiVCr LHEAs were prepared by vacuum arc melting, the phase constitution and microstructure of alloys were characterized, and mechanical properties of alloys were tested. Discussion on the Laves phase in HEAs primarily focused on the following two questions from four perspectives: phase constitution, microstructure, mechanical properties, and solidification. The questions are as follows: (1) Whether the phase selection and competitive nucleation mechanism of the Laves phase be well elucidated? (2) Is it possible to improve the mechanical properties of alloys by effectively controlling Laves phase's volume fraction, and providing guidance for the composite design of high-performance LHEAs?

2 Materials and methods

The Co_xAlNbTiVCr LHEAs (x=0, 0.5, 1, 1.5, and 2, at%) alloys were prepared by vacuum arc melting using the element particles

of Co, Al, V, Cr, Nb, and Ti with high purity (>99.99wt.%), which are referred to as Co0, Co0.5, Co1, Co1.5, and Co2 alloys, respectively. Table 1 shows the nominal composition of the alloys. Table 2 summarizes the physical properties of the above elements, where VEC is valence electron concentration and χ is electronegativity. Raw materials were placed in order of increasing melting point from the top to the bottom of the water-cooled copper crucible. All the button ingots were remelted six times to ensure chemical homogeneity. Specimens with dimensions of 10 mm×9 mm×9 mm were cut from the button ingots by wire cutting for phase and microstructure analysis. The samples were sanded with sandpapers of #400, #800, #1200, and #2000, then polished, and finally subjected to ultrasonic washing with medium alcohol before microstructure observation.

The crystalline structures of alloys were characterized by X-ray diffraction (XRD, Empyrean) with Cu-K α radiation in a 2 θ range from 20° to 100°. Microstructure characterization and elemental analysis of samples were performed using FEI Talos F200x transmission electron microscopy (TEM) with an accelerating voltage of 200 kV. To obtain TEM samples, mechanical grinding was employed to reduce the thickness of the sample to less than 50 µm. The subsequent method used for further thinning the sample was ion thinning. The microstructures of the samples were characterized using scanning electron microscopy (SEM, Merlin Compact) equipped with an energy dispersive X-ray spectrometer (EDS).

The room temperature compressive tests were conducted using a universal testing machine (AG-Xplus) with cylindrical samples (Φ 4 mm×6 mm) at a rate of 0.5 mm·min⁻¹. Vickers hardness tests were carried out under a load of 1.96 N for 15 s

Table 1: Nominal compositions of Co_AINbTiVCr LHEAs (x=0,0.5, 1, 1.5, and 2, at.%) alloys

Alloy	AI	Nb	Ti	v	Cr	Со
Co0	20	20	20	20	20	0
Co0.5	19.9	19.9	19.9	19.9	19.9	0.5
Co1	19.8	19.8	19.8	19.8	19.8	1
Co1.5	19.7	19.7	19.7	19.7	19.7	1.5
Co2	19.6	19.6	19.6	19.6	19.6	2

Table 2: Physical properties of elements in Co_xAlNbTiVCr (x=0, 0.5, 1, 1.5, and 2)

Element	Density (g∙cm⁻³)	Atomic radius (Å)	Shear modulus (GPa)	Molar mass	Melting point (°C)	VEC	χ
Со	8.9	1.26	83	58.93	1,495	9	1.88
AI	2.7	1.43	26	26.98	660	3	1.61
Nb	8.57	1.48	38	92.91	2,468	5	1.6
Ti	4.5	1.45	44	47.90	1,668	4	1.54
V	5.96	1.35	47	50.94	1,890	5	1.63
Cr	7.19	1.27	115	51.99	1,907	6	1.66

on a Vickers hardness tester (FM-700). Fiveteen valid data points were selected for each sample, and the average values after removing the maximum and minimum values were recorded. The actual densities of the alloys were measured using the Archimedes drainage method, and the average of three measurements was taken for each alloy. The solvent for Archimedes drainage method was distilled water.

3 Results

3.1 Phase identification

Figure 1 shows the XRD results of the Co_xAlNbTiVCr (x=0, 0.5, 1, 1.5, and 2) LHEAs. The Co0 LHEA forms only the B2 phase with the space group Im-3m (229). With the addition of Co, diffraction peaks of C14 and C15 Laves phases appear in the Co, AlNbTiVCr LHEAs. According to MDI Jade 6.0 software, the C14-type Laves phase is detected as a hexagonal structure with the space group P63/MMC and the C15-type Laves phase is recognized as a cubic structure with the space group Fd3m (227), which is consistent with the results in Refs. [20, 23, 34]. With further increasing the Co content, the peak intensity of the Laves phases is enhanced. The (110) diffraction peak corresponds to 40.66°, 40.74°, 40.82°, 40.69°, and 40.37° in the Co0, Co0.5, Co1, Co1.5, and Co2, respectively. The lattice parameters of the B2 phase in the Co_xAlNbTiVCr LHEAs were calculated by the Bragg equation: $2d\sin\theta = n\lambda$ (where d is interplanar spacing, *n* is diffraction enhanced order, θ and λ represent respectively the angle between the incident X-ray and the corresponding crystal plane, and the wavelength of the X-ray) and XRD-pdf cards. The lattice constants of Co0, Co0.5, Co1, Co1.5, and Co2 alloys are 0.31552 nm, 0.31347 nm, 0.31209 nm, 0.314522 nm, and 0.31864 nm, respectively. The calculations reveal that the lattice parameters tend to decrease and then increase, influenced by Co doping. The main reason for this tendency



Fig. 1: XRD patterns of Co_xAINbTiVCr LHEAs

can be speculated as follows: as the Co content ranges between 0 and 1, the Co element with a small atomic radius dissolves into the B2 matrix through solid solution, leading to lattice distortion and resulting in solid solution strengthening. When the Co content is between 1 and 2, element Co combines with other elements in the alloy to promote large-scale phase transformations and release lattice distortion energy, which decreases the lattice parameter ^[35].

3.2 Microstructure characterization

Figure 2 shows the microstructure images of $Co_xAlNbTiVCr$ LHEAs with different Co contents. It shows three contrasts, including black, white, and gray. Combined with the XRD results, it can be inferred that the white phase is the C14 Laves phase, the black phase is the C15 Laves phase, and the gray



Fig. 2: SEM micrographs of Co_xAINbTiVCr LHEAs: (a) Co0; (b) Co0.5; (c) Co1; (d) Co1.5; (e) Co2; and (d1) local enlargement of (d)

phase is B2 phase. Co0 alloy exhibits a single B2 phase, while the other alloys exhibit a three-phase structure. Table 3 shows the chemical composition of the constituent phases in Co1.5 alloys. It indicates that the C14 Laves phase is enriched with Nb and Cr elements, the C15 Laves phase is enriched with Co and Ti elements, and the B2 matrix phase is homogeneous in the content of each element, which is consistent with the results of Fig. 3. The volume fractions of C14 and C15 Laves are counted using ImageJ-plus 6.0 software and summarized in Fig. 4. It is noteworthy that volume fractions of two kinds of Laves phases gradually increase with the increase of Co content. The microstructure of the alloys exhibits a dendritic structure with inter-dendritic regions (ID) and dendritic regions (DR) due to constitutional supercooling. Laves phase is preferentially segregated from the ID owing to the relatively higher melting points of the constituent elements of Laves phase.

Figure 5 reveals the TEM-HAADF and SAED patterns of Co1.5 alloy, illustrating that the Co1.5 alloy possesses two types of Laves phase: hexagonal structure C14 Laves phase, as shown in Region 1, and cubic structure C15 Laves phase, as shown in Region 2. The

Table 3: Chemical composition of the constituent phases in Co1.5 alloy (at.%)

Alloy	Phase	AI	Nb	Ti	v	Cr	Co
Co1.5	C14	18.13	22.88	15.89	13.95	24.26	4.89
	C15	17.22	11.89	38.96	9.47	16.12	6.34
	B2	18.96	19.66	18.89	18.51	22.13	1.85

 $[2\overline{110}]$ zone axis SAED image confirms that the Nb-, Cr-rich interdendritic region is C14-type Laves phase. The $[\overline{112}]$ zone axis SAED image proves that the Co-, Ti-rich interdendritic region is a C15-type Laves phase.

3.3 Densities and mechanical properties of Co_xAINbTiVCr LHEAs

Previous research has reported the densities of LHEAs are below 7 g \cdot cm⁻³ ^[36]. In this work, the theoretical density calculation equation is as below ^[37-39]:

$$\rho_{\rm mix} = \frac{\sum_{i=1}^{n} C_i M_i}{\sum_{i=1}^{n} C_i M_i / \rho_i}$$
(1)

where C_i is the atomic concentration, M_i is the relative atomic weight, and ρ_i is the theoretical density of each element in this alloy system. Table 4 lists the theoretical density, actual density, and specific strength of all the alloys in this work. The results show that the theoretical density of all alloys in this work is less than 7 g·cm⁻³, furthermore, the theoretical density is consistent with the actual density, and the alloys have a good consistency.

Figure 6 demonstrates the compressive stress-strain curves of Co_xAlNbTiVCr LHEAs at room temperature. The results show that Co0 alloy has a high room-temperature compressive strength but poor deformation capability, the compressive strength is 1,520.8 MPa and the fracture strain is only 7.1%. Co1.5 alloy exhibits the highest room temperature compressive strength of 1,844.4 MPa, and





Fig. 3: EDS mapping of Al, Nb, Ti, V, Cr, and Co of Co1.5 alloy



Fig. 4: Volume fraction of C14 and C15 Laves phases

the fracture strain reaches the maximum value of 16.4%. In general, this alloy system exhibits poor deformation ability, and the stress-strain curves further show that the alloys fracture before reaching the yield stage during compression. As Co content increases, excessive generation of second phases leads to deterioration of mechanical properties.

When the content of Co increases from 0 to 1.5at.%, the compressive strength of the alloy increases by 21.28%. Specific strength is also an effective index of structural materials. As shown in Table 4, Co1.5 alloy has the best mechanical performance with a maximum specific strength of 320.21 MPa \cdot g⁻¹·cm⁻³. Figure 7 shows the Vickers hardness of Co_xAlNbTiVCr LHEAs. Vickers hardness of the alloys shows the trend to increase and



Fig. 5: TEM-HAADF (a) and SAED patterns of Regions 1 (b) and 2 (c) in (a) of Co1.5 alloy

Table 4	: Theoretical der strength of all a	nsity, actual densit alloys	ty, and specific
	Theoretical	Actual densitv	Specific

Alloy	Theoretical density (g∙cm⁻³)	Actual density (g·cm³)	Specific strength (MPa·g⁻¹·cm⁻³)
Co0	5.73	5.79 ±0.02	265.41
Co0.5	5.74	5.81±0.04	286.12
Co1	5.75	5.83±0.02	309.13
Co1.5	5.76	5.84±0.03	320.21
Co2	5.77	5.85±0.02	283.24



Fig. 6: Compressive stress-strain curves of Co_xAINbTiVCr LHEAs at room temperature

then decrease with the addition of Co. Co1.5 alloy exhibits the highest Vickers hardness of 699.4 HV. Previous work has demonstrated that the improvement in Vickers hardness is mainly associated with an increase in the volume fraction of the C14 Laves phase ^[21, 22].

Figures 8(a) and (b) list the data of mechanical properties based on $Co_xAINbTiVCr$ LHEAs and other HEAs ^[16, 17, 37, 41, 42-48]. According to Fig. 8(a), it is obvious that the $Co_xAINbTiVCr$ LHEAs exhibit excellent mechanical properties. Figure 8(b) shows the comparison of theoretical density and Vickers hardness between Co1.5 alloy and other alloys. The results show that Co1.5 alloy has a higher hardness compared to other alloys.



Fig. 7: Vickers hardness of Co_xAlNbTiVCr



Fig. 8: Mechanical properties based on Co_xAINbTiVCr LHEAs and other HEAs: (a) specific strength and fracture strain; (b) Vickers hardness and theoretical density ^[16, 17, 37, 41, 42-48]

4 Discussion

Why are both the BCC solid solution phase and Laves phase present in the microstructure? Research demonstrates that high configurational entropy promotes the formation of a solid solution phase and hinders the formation of intermetallic compounds ^[49-52]. After the solidification of high entropy alloys, there are three common types of solid solution phases formed: HCP, FCC, or BCC ^[53-64]. In this work, AlNbTiVCr LHEA has a single B2 solid solution. The formation of the solid solution phase in HEA is mainly controlled by mixing enthalpy, mixing entropy, difference in atomic radius size, valence electron concentration value, electronegativity, etc. The common criteria for predicting solid solution formation in high entropy alloys is as follows:

$$\Delta H_{\rm mix} = 4 \sum_{i=1, j \neq i}^{n} C_i C_j \Delta H_{ij}^{\rm mix}$$
(2)

$$\operatorname{VEC} = \sum_{i=1}^{n} C_i (\operatorname{VEC}) i \tag{3}$$

$$\delta = \sqrt{\sum_{i=1}^{n} C_i \left(1 - r_i / \overline{r}\right)^2} \times 100\%$$
(4)

$$\Delta S = -R \sum_{i=1}^{n} C_i \ln C_i \tag{5}$$

$$\Delta \chi = \sqrt{\sum_{i=1}^{n} C_i \left(\chi_i - \overline{\chi} \right)} \,\overline{\chi} = \sum_{i=1}^{n} C_i \chi_i \tag{6}$$

where ΔH_{mix} , VEC, δ , ΔS , and $\Delta \chi$ are mixing enthalpy, valence electron concentration value, atomic radius size, mixing entropy, and electronegativity difference ^[57]. *R* is the ideal gas constant, which is approximately equal to 8.314 J·mol⁻¹·K⁻¹. The C_i and C_j represent the content of the *i*th and *j*th elements, respectively. χ_i represents the electronegativity of the *i*th element. Table 5 shows the calculation results of ΔH_{mix} , VEC, δ , ΔS_{mix} , and $\Delta \chi$ for Co_xAlNbTiVCr LHEAs. The well-known standard for the formation of HEA solid solution phases is $\Delta S_{\text{mix}} > 1.5R$, -15 kJ·mol⁻¹ $<\Delta H_{\text{mix}} < 5$ kJ·mol⁻¹, and $\delta \le 6.6\%$. B2 phase is considered to be stable under a low VEC (<6.87) conditions ^[54]. In summary, all HEAs in this work meet the conditions for the formation of a solid solution phase and the solid solution type is BCC-type.

Besides the B2 phase, the Laves phase also exists in the microstructure, which is attributed to the fact that high mixing entropy cannot thoroughly prevent the contribution of mixing enthalpy in free energy. Mixing enthalpy is also an important factor that promotes the intermetallics. Table 6 lists mixing enthalpies by Miedema's model in this alloy system. The negative mixing enthalpy indicates a stronger mutual attraction between elements, which is more conducive to the formation of the Laves phase. Table 6 shows the mixing enthalpy between Co and Al, Nb, Ti, V, and Cr are -19 kJ·mol⁻¹, -25 kJ·mol⁻¹, -28 kJ·mol⁻¹, -14 kJ·mol⁻¹, and -4 kJ·mol⁻¹, respectively. Co has the most negative mixing enthalpy with Ti, which indicates the strongest mutual attraction between Co and Ti.

Furthermore, the electronegativity of Co is 1.88, which is the maximum in the Co_xAlNbTiVCr alloy system. Co and Ti elements have the largest electronegativity difference, as shown in Table 2. The larger the electronegativity between elements, the easier to form an intermetallic compound phase^[57]. According to the above analysis, the most negative mixing enthalpy and maximum electronegativity difference between Co and Ti contribute to the formation of the C15 type Laves phase. In this alloy system, elements with relatively

Table 5: Calculation results of ΔH_{mix} , VEC, δ , ΔS_{mix} and $\Delta \chi$ for Co_xAINbTiVCr LHEAs

Alloy	∆ <i>H</i> _{mix} (kJ·mol⁻¹)	VEC	δ (%)	∆S _{mix} (J·mol ⁻¹ ·K ⁻¹)	Δχ
Co0	-14.56	4.600	5.47	13.38	0.381
Co0.5	-14.69	4.622	5.81	13.57	0.369
Co1	-14.82	4.644	5.83	13.71	0.367
Co1.5	-14.95	4.666	5.85	13.83	0.365
Co2	-15.08	4.688	5.87	13.93	0.363

Table 6: Mixing enthalpies (kJ·mol⁻¹) by Miedema's model in this alloy system

Element	AI	Nb	Ti	V	Cr	Co
AI	-	-18	-30	-16	-10	-19
Nb		-	2	-1	-7	-25
Ti			-	-2	-7	-28
V				-	-2	-14
Cr					-	-4
Со						-

high melting points include Nb and Cr. Whereas Nb and Cr elements possess a significant difference in atomic radius size. The atomic radius ratio of A and B in the AB₂ type Laves phase is between 1.1 and 1.6, and the atomic radius ratio of Nb and Cr is 1.165, which is suitable for forming the Laves phase ^[29]. The melting point of alloys can be expressed as Eq. (7):

$$T_{\rm m} = \sum C_i (T_{\rm e})_i \tag{7}$$

where C_i is element content, T_m is the melting point of alloys,

and $T_{\rm e}$ represents the element melting point. Melting points of Co0, Co0.5, Co1, Co1.5, and Co2 alloys are calculated to be 1,718.60 °C, 1,717.48 °C, 1,716.36 °C, 1,715.25 °C, and 1,714.13 °C, respectively, indicating the doping of Co element slightly reduces the melting point of the alloys.

Figure 9 presents the solidification mechanism diagram of Co1.5 alloy. Due to the fact that arc melting is a rapid solidification process in a non-equilibrium state, the melting point is not the only controlling factor for phase precipitation during the solidification process. The mixing enthalpy between Co and Ti is the most negative, so the C15 Laves phase rich in Co and Ti preferentially precipitates during the solidification process. As the temperature decreases, a large amount of C14 Laves phase rich in Nb and Cr precipitates. The C15 Laves phase may supply heterogeneous nucleation sites. Therefore, the C14 Laves nucleates and grows around C15 Laves phase.

Figure 10 exhibits the compression fracture morphology of $Co_xAINbTiVCr$ LHEAs. The oberserved fracture morphology does not reveal any dimples. Instead, the fracture surface exhibits many river patterns and cleavage steps. These features are characteristic of a cleavage fracture, namely the brittle fracture. In particular, the presence of Laves phase increases strength and hardness of the alloys, while also increasing its deformability.



Solidifying

Fig. 9: Solidification mechanism diagram of Co1.5 alloy



Fig. 10: Compression fracture morphologies of Co_xAlNbTiVCr LHEAs: (a) Co0; (b) Co0.5; (c) Co1; (d) Co1.5; and (e) Co2

5 Conclusions

In this study, it is found an increased concentration of Co could effectively improve mechanical properties of $Co_xAINbTiVCr$ LHEAs primarily through solid solution strengthening due to lattice distortion and second phase strengthening due to the formation of the Laves phase. After a series of characterizations, the following conclusions are found:

(1) AlNbTiVCr LHEA is composed of a single B2 phase, while the mixture of B2 phase, C14 Laves phase, and the C15 Laves phase is observed in the Co_xAlNbTiVCr LHEAs (x=0.5, 1, 1.5, 2).

(2) The calculation results of different phase formation prediction parameters are consistent with the experimental results. With the addition of Co, the C14 Laves phase and the C15 Laves phase continuously appear, and their volume fraction shows an increasing trend in varying degrees.

(3) In this work, Co1.5 alloy exhibits the best mechanical properties with a compressive strength of 1,844.4 MPa, and a specific strength of 320.21 MPa \cdot g⁻¹·cm⁻³. Co1.5 alloy possesses the maximum Vickers hardness of 699.4 HV. The improvement of mechanical properties of alloys comes from solid solution strengthening and the second phase strengthening caused by two types of Laves phases: C14 and C15.

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Conflict of interest

Prof. Rui-run Chen is an EBM of *CHINA FOUNDRY*. He was not involved in the peer-review or handling of the manuscript. The authors have no other competing interests to disclose.

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