A new single crystal high entropy alloy with excellent high-temperature tensile property

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Abstract

A new single crystal high entropy alloy is developed and produced using Bridgman directional-solidification and seed-crystal method. The microstructure exhibits the directional dendrite morphology and is mainly composed of the FCC matrix and the L1\textsubscript{2} ordered precipitates. Its high-temperature yield strengths are far higher than those of the first-generation single crystal superalloys, and close to those of the second-generation ones. The solid-solution strengthening effect from the high content of W and Mo, as well as the precipitation strengthening effect from the combined addition of Al, Ti, Ta, and Nb, should be responsible for the excellent high-temperature strength of the investigated alloy.

1. Introduction

Compared with the traditional alloys with one or two principal matrix elements, high entropy alloys (HEAs) are composed of multiple elements [1]. The ratio of component elements has transformed from equal atom ratio to unequal atom ratio compositions, and the phase compositions of HEAs have also gradually transited from single-phase solid solution to multi-phase alloys [2]. The HEAs usually have excellent performance, such as excellent ductility [3], high strength [4], corrosion resistance [5], and radiation resistance [6]. The HEAs can be classified into three alloy families. These families include the transition metal HEAs (such as NiCoCrFeAl\textsubscript{4}x [7] and (NiCoCr)\textsubscript{94}Al\textsubscript{3}Ti\textsubscript{3} [8]), the refractory metal HEAs (such as HfMo\textsubscript{3}NbTaTiZr [9] and NbTaWMoSi\textsubscript{4} [10]), and the light-weight metal HEAs (such as AlFeMgTiZn [11] and Al\textsubscript{2}CuMgSnTiZn\textsubscript{7} [12]).

There are some transition metal HEAs which are designed to obtain coherent L1\textsubscript{2} precipitates (γ\textsuperscript{'} phase) via the addition of Al and Ti elements into the FCC matrix (γ phase), which always have good mechanical properties [8, 13–18]. Liu \textit{et al} [13] studied the effect of Al addition on microstructure and mechanical properties of Ni\textsubscript{70−x}Co\textsubscript{15}Cr\textsubscript{15}Al\textsubscript{x}. When x = 12.5 and 17.5, they show the γ + γ\textsuperscript{'} dual-phase structure and have excellent compressive yield strength at room temperature (RT). Liang \textit{et al} [14] prepared Ni\textsubscript{2.5}Cr\textsubscript{0.5}FeAl\textsubscript{0.5}V\textsubscript{0.2} alloy by adding Al and V elements to the NiCrFe matrix. The RT tensile yield strength and ultimate strength of this alloy are 1570 MPa and 1763 MPa, respectively. Yang \textit{et al} [15] obtained the (NiCoFe)\textsubscript{94}Al\textsubscript{7}Ti\textsubscript{2} by adding Al and Ti elements to the NiCoFe. The RT tensile yield strength of this alloy is about 1 GPa, which is five times higher than that of the single-phase NiCoFe alloy. Zhao \textit{et al} [16] designed a (NiCoCr)\textsubscript{94}Al\textsubscript{7}Ti\textsubscript{3} alloy containing γ\textsuperscript{'} precipitate with an outstanding RT tensile strength. Zhang \textit{et al} [16] prepared Ni\textsubscript{47.7}Co\textsubscript{17.5}Cr\textsubscript{17.5}Fe\textsubscript{9.3}Al\textsubscript{7.5}Ti\textsubscript{2}Mo\textsubscript{1} alloy by adding solid solution strengthening element Mo. Its RT tensile yield strength is higher by 5% than that of Ni\textsubscript{48.7}Co\textsubscript{17.5}Cr\textsubscript{10}Fe\textsubscript{9.3}Al\textsubscript{7.5}Ti\textsubscript{2} alloy with little loss of plasticity. Ni\textsubscript{47.9}Co\textsubscript{16.8}Cr\textsubscript{7.7}Fe\textsubscript{8.9}Al\textsubscript{10.2}Ti\textsubscript{3.6}Nb\textsubscript{1.2}Mo\textsubscript{0.9}W\textsubscript{0.4}C\textsubscript{0.4} (hereinafter named Tsao alloy) is designed to demonstrate better strength at high temperatures with the addition of Nb and W [17]. The tensile yield strength of the...
directionally solidified Tsao alloy is 476 MPa at 950 °C, which is close to that of single crystal (SC) superalloy CMSX-2 [18].

However, most studies on the mechanical properties of HEAs are focused on RT properties to date. To obtain the excellent mechanical properties at high temperature, a new HEA was developed based on the above-mentioned Tsao alloy. Compared with the composition of the Tsao alloy, the content of Mo and W is increased, the element of Fe is removed since the Mo and W could produce the more appreciable solid solution strengthening [19]. In addition, the γ′ forming element Ta is added. On the one hand, it can increase the volume fraction of γ′ phase. On the other hand, it would enhance entropy effects of the γ′ phase and further increase the alloy strength [1, 19]. Meanwhile, the contents of Ti and Nb have been reduced in order to avoid the formation of harmful TCP phases. Therefore, the Ni56.11Co16Cr8Al10Ti3.2Ta1.7Nb0.65Mo1.3W2.7C0.34 (hereinafter named HSHEA) is developed herein. It should be emphasized that the SC HSHEA was prepared since the disappearance of grain boundary can increase the strength. The microstructure and high-temperature tensile properties of SC HSHEA were studied. The investigation shows that the HSHEA exhibits the excellent high-temperature performance.

2. Materials and method

All raw materials (Ni, Co, Cr, Al, Ti, Ta, Nb, Mo, W, and C) with purity higher than 99.9 wt% were melted into ingots using a vacuum induction melting furnace. Then, they were prepared into an Φ 7 mm × 110 mm SC rods via the directional-solidification and seed-crystal methods [20]. The samples were heat treated at 1200 °C/10 h/AC + 1080 °C/3 h/AC + 850 °C/24 h/AC, which is adjusted based on existing papers [18]. The tensile test was carried out at 850, 950, and 1000 °C at a strain rate of 10−3 s−1. The gauge diameter and length of samples were 3 and 15 mm, respectively, which depends on required size of the test equipment (figure 1). The tensile-sample surface was ground on #240, #400, #800, #1200, and #2000 SiC emery papers in sequence, before wet-polished.

The heat-treated samples were processed by standard metallographic preparation method. The corrosion agent used is 4 g CuSO4 + 10 ml HCl + 10 ml H2O. The microstructures were analyzed using optical microscopy (OM), scanning electron microscope (SEM), and transmission electron microscopy (TEM). Deformation structures on the uniform strained regions of fractured tensile specimens were characterized by TEM. TEM foils were prepared by a twin-jet polisher with a solution of 10% HClO4 + 90% CH3OH at −30 °C.

3. Results

3.1. Microstructures

The longitudinal and cross section micrographs show that the crystal grows as the dendritic pattern (figures 2(a), (b)). The dendritic region of the HSHEA shows that the precipitates are homogenously distributed in the matrix (figure 2(c)). In addition to the coarse γ′ particles, there are a few γ/γ′ eutectic and bulk phases in the inter-dendritic region (figure 2(d)). The TEM analysis shows that the bulk phases are the MC carbide (figure 3). The analysis of the bulk phases is given below. Based on the statistical analyses, the average diameters of the γ′
particles in the dendritic and interdendritic region of the heat-treated HSHEA are approximately 273 and 709 nm, respectively (figures 2(e) and (f)).

In order to further characterize $\gamma'$ particles and the bulk phases in the interdendritic region, the microstructures of the HSHEA were further observed by TEM (figure 3). There are nearly spherical $L_1_2$ strengthening phases dispersing in the FCC matrix (figure 3(a)), confirmed by the diffraction pattern (inset in figure 3(a)). The high-resolution TEM of these phases are shown in figure 3(b). The fast Fourier transform (FFT) and inverse fast Fourier transform (IFFT) images corresponding to the red and yellow regions are shown in figures 3(c) and (d), which confirmed that they are $\gamma$ and $\gamma'$ phases, respectively [14]. The lattice constants of $\gamma$ and $\gamma'$ phases are 0.3651 nm and 0.3665 nm, respectively, as measured by the FFT images. The lattice misfit between the $\gamma$ and $\gamma'$ phase, $\varepsilon$, is about 0.015%, which is obtained according to the equation,

$$
\varepsilon = \frac{2(a_{\gamma'} - a_{\gamma})}{(a_{\gamma'} + a_{\gamma})}
$$

where $a_{\gamma}$ and $a_{\gamma'}$ are the lattice constants of $\gamma$ and $\gamma'$ phase, respectively. Figure 3(e) shows the interface between $\gamma$ and $\gamma'$ phases, indicating that they are completely coherent. The chemical compositions of the bulk phases based on TEM-EDS were shown in table 1. It indicates that the bulk phases are rich in Ti and Ta, similar to carbide MC (M are usually Ti and Ta) in superalloys [21]. Combined with the diffraction pattern in figure 3(f), we conclude that the bulk phase is (Ti, Ta)C carbide.
3.2. Tensile property and deformation behavior

Figure 4 are the tensile true stress-strain curves of the HSHEA at different temperatures. Its yield strengths are 1025, 738, and 578 MPa at 850, 950, and 1000 °C, respectively. They gradually decrease as the temperatures increases.

We compared the tensile yield strengths of the HSHEA with the first (PWA1483 [22], DD8 [23], SRR99 [24], DD3 [25], DD13 [26], and CMSX-2 [27]), the second (DD5 [28] and DD 6 [23]) generation Nickel-based SC superalloys and the Tsao alloy [18] at elevated temperatures (figure 5). It should be noted that there are only partial tensile data of the reference alloys in the existing literatures. Firstly, the yield strength of the HSHEA is about 262 MPa higher than the Tsao alloy at 950 °C. Secondly, the high-temperature performance of HSHEA far
exceeds that of the first-generation SC superalloy. For example, its strength is higher than PWA1483 by 32%, 17%, and 62% at 850, 950, and 1000 °C, respectively. The strength of PWA1483 is at a high level in the first-generation Nickel-based SC superalloys since the contents of the γ′ forming elements (Al + Ti + Ta + Nb) and the solid-solution strengthening element (W + Mo) are higher than those of most first-generation SC superalloys (figure 6). Thirdly, the strength level of the HSHEA is also close to those of the second-generation SC superalloys on the condition without adding Rhenium. Therefore, the high entropy alloy designed in present study possesses the excellent high temperature performance.

Figure 4. The tensile true stress-strain curves of the HSHEA at high temperatures.

Figure 5. Comparison of the yield strengths of the HSHEA, Tsao alloy, and SC Ni-based superalloys at different temperatures.

Figure 7 shows the dislocation morphology near the fracture of the HSHEA at different temperatures. At 850 °C (figures 7(a), (b)), there are many superdislocations and the stacking faults (SFs) in γ′ particles. The movements of superdislocations are accompanied by the formation of antiphase-boundary (APB) [29–31]. The SF formation is achieved by the interfacial dislocations decomposing into the γ′ phase in the following manner: \(1/2[011] + 1/2[110] \to 1/3[1\overline{2}1] + 1/6[1\overline{2}1]\) [32–34]. The 1/6[1\overline{2}1] dislocations remain at the interface, and the 1/3[1\overline{2}1] dislocations cut through the γ′ particles to form SFs. At 950 °C (figure 7(c), (d)), in addition to the little quantity of the APB and SF shearing, there appear many curved and loop dislocations, which indicate that the dislocations mainly moved via the cross-slip and Orowan bypass mechanisms at matrix. At 1000 °C (figures 7(e), (f)), there is no cross-slip or SF. Many wavy dislocations are arranged along the γ/γ′ interface. This is the result of the combination of dislocations slipping and climbing [32]. The APB shearing also occurs at the temperature. Moreover, the fine γ′ phase precipitates in the deformation.
4. Discussion

The results show that the HSHEA is mainly composed of the FCC matrix and the L1₂ ordered precipitates. It exhibits excellent high-temperature tensile properties. This should be related to its compositions, which affect the phase strength by the solid-solution strengthening and the precipitation strengthening and further improve the mechanical property. Compared with the compositions of the Tsao alloy and the first-generation SC superalloys, those of the HSHEA possess the following characteristics (figure 6): (1) The combined content of the solid-solution strengthening element W and Mo is almost highest. The solid-solution strengthening coefficients of W and Mo are much higher than those of the other solid-solution elements, such as Co, Cr, and Fe (table 2) [19]. (2) The more types of γ’ forming elements were added, including Al, Ti, Ta, and Nb. The most first-generation SC superalloys only have two or three types of γ’ forming elements. For example, Al, Ti, and Ta are
added to the PWA1483 and DD408. Al, Ti, and Nb are done to the Tsao alloy. Obviously, as the number of element types increases, the mixed entropy of the γ′ phase increases [35]. The high mixed entropy will give rise to severe lattice distortions and slow diffusion of elements [36, 37]. Of course, the γ′ forming elements also play some roles in the solid-solution strengthening. The role of elements could be embodied in the dislocation morphology and the microstructure change during the tensile deformation.

As is well-known, the strength of alloy is controlled by the dislocation movements. To some extent, the more difficultly the dislocation moves, the higher strength the alloy possesses. In the γ′ precipitation-strengthening alloys, the dislocations originate the γ matrix channel, moves to the interface, and finally go into the γ′ particles [38]. The element addition could have effects on the dislocation movements in both γ and γ′ phases [38]. Firstly, the effect of solid-solution strengthening elements on the dislocation is discussed. The solid-solution elements give rise to severe lattice distortion and form a large stress field, which hinders the dislocation motion. The atomic radius of Mo and W is about 10% larger than that of Cr and Co, which is reflected in the coefficient summary in table 2. On the one hand, the solid-solution elements significantly reduce the SF energy, which will make the cross-slip perform difficulty and reduce the dislocation movement ability [39]. It can improve the strength of the alloy. Xie et al [40] showed that the number of Ni stacking faults decreases by the element addition as follows:

\[
\Delta \gamma \text{SF} = 1.66(\text{at\% Cr}) + 1.72(\text{at\% Al}) + 8.0(\text{at\% Ti}) + 1.66(\text{at\% Mo}) + 0.96(\text{at\%. Co}).
\]

The Nb and Ta are ignored because of their low content. Johnson et al [41] studied the influence of W on SF energy in the Ni - W system. It is found that initially W drastically decreases the SF energy and that the effect then saturates. The SF energy of Ni - W systems decreases by about 40% and 60% when 1 ~ 2 at% and >4 at% W, respectively is added [41, 42]. The influence of W on SF energy in the Nickel-base alloy can be estimated. Therefore, W and Mo reduces more SF energy than Co and Cr [43, 44]. Based on the above data, the decrease in SF energy in γ phase of the HSHEA and the first-generation SC crystal is summarized in table 3. This fully demonstrates that the large SF energy of the investigated alloy is reduced with the W and Mo addition compared to that of the other alloys with slightly low W and Mo content. Therefore, it can be concluded that the large content of W and Mo effectively contributes on the strength improvement of the investigated alloy.

Secondly, there are two ways that dislocations propagate from the γ matrix into the γ′ phase in the γ′ precipitation-strengthening alloys [45]. One is shearing of the a/2[110] dislocation in the γ′ phase, which creates an APB on its movement path. The other is the formation of SF in the γ′ phase. The more APB energy, the more pronounced strengthening. The substitution of Ti, Ta, and Nb at the Al site in the γ′ phase could increase the APB energy [46]. Accordingly, the content of Ti, Ta, and Nb determines the increasing magnitude of APB energy. All the three elements are added in the investigated alloy, while one or two types are done in the reference alloys. The quantity of the three elements in the investigated alloys is relatively larger (table 4). The contents of Ta and Nb is largest, which increase the more magnitude of APB energy than the element Ti [44]. Thus, the APB energy in the HSHEA is relatively bigger. In addition, the addition of W has the excellent solid-solution strengthening effect on the γ′ phase (table 2). This will hinder the dislocation shearing of the γ′ phase. The APB shearing were observed within all the investigated temperature (figure 6), specifically at 1000 °C. However, it

| Table 2. Solid-solution strengthening coefficients for the γ and γ′ phases for each element [19]. |
|---|
| Element | Cr | Co | Mo | W | Nb | Al | Ti | Ta |
| Coefficient in γ (MPa/α1/2) | 337 | 39.4 | 1015 | 977 | 1183 | 225 | 775 | 1191 |
| Coefficient in γ′ (MPa/α1) | 11 | — | 41.88 | 40 | 56 | — | 18.3 | 78.33 |

| Table 3. Composition and the decrease in SF energy of γ matrix of the HSHEA and the first-generation SC superalloys (at%). |
|---|
| Alloy | Ni | Co | Cr | Al | Ti | Ta | Nb | Mo | W | ΔγSF% |
| HSHEA | 43 | 24.7 | 21 | 2.8 | 0.5 | 0 | 0 | 3.3 | 4.8 | 133.1 |
| PWA1483 | 57.1 | 17.6 | 27.8 | 0.6 | 0.06 | 0.04 | 0 | 1.3 | 1.6 | 106.7 |
| DD8 | 53.5 | 13.9 | 29.3 | 1 | 0.04 | 0 | 0 | 0 | 2.2 | 104 |
| DD3 | 56.9 | 10.8 | 27.9 | 1.6 | 0.02 | 0 | 0 | 1.8 | 0.9 | 102.7 |
| SRR99 | 55.3 | 11.9 | 28.8 | 1.3 | 0.02 | 0.009 | 0 | 0 | 2.7 | 101.6 |
| DD13 | 51.7 | 17.6 | 27.6 | 0.8 | 0.06 | 0.03 | 0 | 2.2 | 0 | 68.2 |
| CMSX-2 | 56.2 | 11.7 | 27.7 | 1.4 | 0.01 | 0.02 | 0 | 0.5 | 2.4 | 100.6 |

The compositions of HSHEA are obtained by TEM-EDS. The compositions of superalloys are calculated by the JmatPro software.
could not appear in the most first-generation Ni-based superalloys during the tensile deformation at 1000 °C [30, 47]. This indicates the γ′ phase still exhibits the high strength at this temperature in HSHEA, which is just because of its high APB energy.

The formation of the SF is determined by the SF energy, which is related with the temperature and the alloying elements [33, 48, 49]. As the SF energy increases, it is difficult to form SFs. The SF energy gradually increases with the temperature increases [33]. Thus, the quantity of SF gradually decreases with the temperature increasing and it finally disappears. The SF energy would be decreased by adding the alloying elements [48], especially W [41]. Therefore, the alloying elements could increase the temperature when the SF disappear in the Nickel-based superalloys during the tensile deformation. For example, the W content of the M4706 [32] and N3 [40] alloy is 4.6 and 13 ~ 15.5 wt%, respectively. The other-element contents of the two alloys are almost same. The temperature when the SFs disappear during the tensile deformation for the three alloys is above 900 and 1000 °C, respectively. It is well-known that the SF existence indicates the difficulty of the dislocation movement. In other words, the higher temperature the SF disappears, the stronger strength the alloy would possess. The temperature when the SF disappears in the HSHEA in our study is higher than that in the PWA1483 [22]. This just indicates the strength of the HSHEA should be much more than that of the PWA1483, and in fact it does. It is also consistent with the composition’s characteristic of the two alloys, in which the W content is 2.7 and 1.2 at% with the almost same contents of other elements.

Finally, the fine γ′ particles precipitate in the deformation of the HSHEA at 1000 °C, which could be related to the low lattice mismatch between the γ and γ′ phases. The lattice parameters of the γ and γ′ phases with different temperatures can be expressed as [50, 51]:

\[
a_\gamma = 3.524 + (1.79C_{Al}^\gamma + 0.196C_{Co}^\gamma + 1.10C_{Cr}^\gamma + 4.78C_{Mo}^\gamma + 7.0C_{Ta}^\gamma + 4.44C_{W}^\gamma + 4.22C_{Ti}^\gamma + 7.0C_{Nb}^\gamma) \times 10^{-3} + 5.741 \times 10^{-5} \times (T - 25) - 1.010 \times 10^{-9} \times (T - 25)^2 \text{ (Å)}
\]

\[
a_{\gamma'} = 3.57 + (5.0C_{Ta}^{\gamma'} + 4.6C_{Nb}^{\gamma'} - 0.04C_{Cr}^{\gamma'} + 2.08C_{Mo}^{\gamma'} + 1.94C_{W}^{\gamma'} + 2.58C_{Ti}^{\gamma'}) \times 10^{-3} + 6.162 \times 10^{-5} \times (T - 25) - 1.132 \times 10^{-8} \times (T - 25)^2 \text{ (Å)}
\]

where \(a_\gamma\) and \(a_{\gamma'}\) are the lattice parameters of the γ and γ′ phases, respectively, \(C_i^\gamma\) and \(C_i^{\gamma'}\) are the content of element \(i\) in the γ and γ′ phases, respectively, \(T\) is temperature. The chemical compositions of γ′ phases of HSHEA are characterized based on EDS, and those of other alloy are calculated by the JmatPro software. Based on the equations (2) and (3), the lattice misfit of the alloys is shown in Table 5. It can be seen that the lattice misfit of the investigated alloy is smallest. This is due to the W segregation in the γ′ phase, which increases its lattice constant. The lower lattice misfits reduce the nucleation barrier of precipitation phase, which drive the fine γ′ particles to precipitate during the deformation. The same phenomenon was reported in the deformation of the high W-content Nickel-based superalloy [40]. Owing to their high-density and dispersed nanosize, the γ′ particles effectively strengthen the HSHEA. In addition, the low mismatch stabilizes the coherent interface by reducing the elastic strain energy, which is beneficial for reducing the roughening driving force [52]. It is beneficial for the improvement in the high-temperature strength of the alloy.

### Table 4. The content of Al, Ti, Ta, and Nb in the HSHEA, Tsao alloy, and the first-generation SC superalloys (wt.%).

| Alloy      | Al   | Ti | Ta | Nb | Ti + Ta + Nb | Ta + Nb |
|------------|------|----|----|----|--------------|---------|
| HSHEA      | 4.4  | 2.5| 5.1| 1.0| 8.6          | 6.1     |
| Tsao alloy | 5.0  | 5.0| 0.0| 2.0| 7.1          | 2.0     |
| PWA1483    | 3.5  | 4.0| 4.9| —  | 8.9          | 4.9     |
| DD8        | 3.9  | 3.9| 1.0| —  | 4.9          | 1.0     |
| SRR99      | 5.5  | 2.2| 3.0| —  | 5.2          | 3.0     |
| DD3        | 5.9  | 2.0| —  | —  | 2.0          | 0.0     |
| DD13       | 4.0  | 4.2| 5.2| —  | 9.4          | 5.2     |
| CMSX-2     | 5.6  | 1.0| 6.0| —  | 7.0          | 6.0     |

### Table 5. The misfit of the HSHEA and some first-generation SC superalloys at 1000 °C.

| Alloy | HSHEA | PWA1483 | DD8 | DD3 | SRR99 | DD13 | CMSX-2 |
|-------|-------|---------|-----|-----|-------|------|--------|
| \(a_\gamma\) (Å) | 3.6509 | 3.6276 | 3.6259 | 3.6275 | 3.6273 | 3.6253 | 3.6278 |
| \(a_{\gamma'}\) (Å) | 3.6554 | 3.6545 | 3.6424 | 3.6326 | 3.6392 | 3.6524 | 3.6411 |
| \(\varepsilon\) (%) | 0.12  | 0.74   | 0.45  | 0.14 | 0.33  | 0.74  | 0.36   |
In summary, the high content of W and Mo causes the large lattice distortion and dramatically reduce the SF energy, thereby enhancing the solid-solution strengthening effect. The combined addition of the $\gamma'$ phase-strengthening elements, such as Al, Ti, Ta, and Nb, effectively strengthens the $\gamma'$ phase by increasing the APB energy. The composition characteristic of the designed HSHEA makes it exhibit the excellent high-temperature tensile property.

5. Conclusion

A new high entropy alloy has been designed. The microstructure and the high-temperature tensile behavior is studied. The main conclusions are as follows:

(1) The SC HSHEA directionally solidifies as the dendrite morphology. The dendrite arm and interdendrite regions are mainly composed of the FCC matrix and the $L1_2$ ordered precipitates. There are a small amount of eutectic and carbide MC in interdendritic regions.

(2) The tensile yield strengths of the HSHEA are 1025, 738, and 578 MPa at 850, 950, and 1000 $^\circ$C, respectively. It shows the excellent high temperature mechanical properties, which is higher than those of the first-generation SC superalloys, and close to that of the second-generation SC ones.

(3) The excellent high-temperature strength of the investigated alloy can be attributed to the solid solution strengthening effect from the high content of W and Mo, as well as the precipitation strengthening effect from the combined addition of Al, Ti, Ta, and Nb.

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Data availability

The data that support the findings of this study are available from the corresponding author on reasonable request.

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