Original Article

Characterization of Y-bearing particles in CNS-I-ODS steel fabricated by vacuum induction melting & casting technique

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ABSTRACT

Vacuum induction melting (VIM) & casting is not a common but a promising approach to produce ODS steel with large volume and high throughput. Meanwhile, the Y-bearing particles in the cast ODS steel presented various chemical compositions and wide size distribution. Thus it is important and necessary to clarify the Y-bearing particle characteristics in the cast ODS steel. To determine the distribution characteristics, chemical compositions, crystal structure and size distribution of the Y-bearing particles, CNS-I-ODS was fabricated by adding 1.0 wt.% metallic yttrium into the molten China Nuclear Steel (CNS) during VIM. The results indicated that some banding structures comprised of yttrium oxides and Y-bearing intermetallic compounds formed along rolling direction. XRD, SEM, TEM and EDS demonstrated that yttrium oxides with the size of 550–900 nm were consisted of Y2O3 and Y(O, C) phases while Y-bearing intermetallic compounds with the size of 26–500 nm were consisted of Fe-Y and Y-W-Ta phases. Besides, Y addition also facilitated the formation of δ-ferrite, refinement of prior austenite grains and precipitation of carbides. Furthermore, it is acceptable that the CNS-I-ODS exhibited similar elongation of 22.7–23.5%, slightly lower strength of 622 MPa and higher ductile-brittle transition temperature of −42 °C compared to the Y-free steel, although some large yttrium oxides were distributed in the former matrix.

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1. Introduction

Oxide-dispersion-strengthened (ODS) ferrite/martensitic (F/M) steels have been widely considered as the candidate materials for advanced fission reactor’s cladding and fusion reactor’s blanket due to the excellent irradiation resistance and high temperature creep properties [1,2]. The popular fabrication route for ODS steel is powder metallurgy (PM), which involves gas atomization to produce a prealloyed powder, mechanical alloying (MA) with fine oxide powders, consolidation, and finally thermomechanical treatment (TMT) [3,4]. Therefore, the PM process is complex and expensive, and the fitness for scaling up to the industrial scale is limited [5]. Thus some alternative fabrication routes for ODS steels were developed and summarized by Bergner et al. as following [6]: I) modified PM route to avoid MA, II) liquid metal (LM) route, III) hybrid route based on elements from both the PM and LM routes, IV) physicochemical route focusing on the fabrication of model alloys. Among them, LM route is suggested to be a promising approach to produce ODS steel with large volume and high throughput.

Han et al. directly added 2 µm Y₂O₃ powders into the molten Fe alloy for producing ODS steels via vacuum induction melting (VIM), magnetic stirring casting, forging and heat treatment [7,8]. The results indicated that some micro-scale Y₂O₃ particles suffered coarsening and agglomeration due to the high interface energy of Y₂O₃ and poor wettability between Y₂O₃ and molten Fe [9]. In order to avoid agglomeration of Y₂O₃ particles, low-concentration, low-viscosity Y₂O₃ colloidal powder suspensions were prepared by Verhiest et al. [10]. Then both the colloidal Y₂O₃ and metallic yttrium (Yₚ) were added into the melt T91 using the rapid solidification processing technique [11]. The results indicated that homogeneously dispersed ODS matrix was achieved in the laboratory cast ingot of T91-Yₚ-Y₂O₃ with the total weight of 60 g as the weak wettability between Y₂O₃ and liquid Fe-Cr alloy can be improved by the addition of Yₚ, which demonstrated the feasibility of ODS steel fabrication by melting and casting technique. Similarly, Yₚ was added into the molten steel during VIM to prepare the ODS F/M steel in refs [12–14] and multiple phases including Y-O, Fe-Y, Fe-Cr-Y, Fe-Y-C, Y-Ti-O, Y-O-Ta-W, Y-O-Ta-C, Y-O-Ta-W-C, Y-Ti-O-Ta-W, Fe-Cr-Ta-Y-S-O with the size ranging from 50 nm to 10 µm were generated. Obviously, Y-bearing particles formed in the F/M matrix presented complex chemical compositions and wide size distribution, which should be further explored and optimized in long-term experiments.

By now, our group has produced China Nuclear Steel (CNS) with excellent mechanical properties, corrosion performance and irradiation resistance in refs [15–21] and CNS-I-ODS (a 9Cr-ODS steel) via in-situ reaction between elemental yttrium in molten Fe and activated oxygen from FeO₂O₃ oxygen carrier during the solidification in ref [22]. Recently, the 12Cr F/M steel (CNS-II) with 0.1 wt%, 0.2 wt% and 0.3 wt% theoretical Y additions were fabricated by VIM & casting method successfully [23]. In the present study, we try to fabricate the CNS-I-ODS by adding Yₚ into the molten steel via VIM & casting method and the aim of this study is to determine the distribution characteristics, chemical compositions, crystal structure and size distribution of the Y-bearing particles. Previous literature proved that the yields of Y during VIM & casting were about 15.4–34.4% as Y additions were 0.091–0.553 wt.% in the 0.3C-1.5Si-1.8Mn-0.083Al steel [24]. Thus 1.0 wt.% Y will be added theoretically in the molten steel to achieve the measured content of about 0.15–0.35 wt.% which is the popular content for ODS steel fabricated via PM [25]. Besides, the Y-free steel, that is, CNS-I will be also prepared via the same method for comparison.

2. Experiments

2.1. Material preparation

Nominal chemical compositions of the CNS-I and CNS-I-ODS are listed in Table 1. Both steel ingots were prepared by VIM & casting method. 100 kg pure irons and alloying elements
with high purity (>99.9%) were induction melted and stirred continuously using an electro-magnetic stirrer under the protective argon atmosphere firstly. Meanwhile, 0.5 kg metallic Y powders with 99.9% purity were placed at the bottom of one mould before casting and the other mould was empty. Then the liquid steel was poured into the two moulds and solidified rapidly from 1600 °C to room temperature (RT) under the protective argon atmosphere. Afterwards, both 50 kg ingots

### Table 1 – Nominal chemical compositions of the CNS-I and CNS-I-ODS, wt.%.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Fe</th>
<th>C</th>
<th>Cr</th>
<th>Mo</th>
<th>W</th>
<th>Mn</th>
<th>Si</th>
<th>V</th>
<th>Ta</th>
<th>Ti</th>
<th>N</th>
<th>Y</th>
</tr>
</thead>
<tbody>
<tr>
<td>CNS-I</td>
<td>Bal.</td>
<td>0.1</td>
<td>9.0</td>
<td>0.4</td>
<td>1.2</td>
<td>0.5</td>
<td>0.15</td>
<td>0.25</td>
<td>0.2</td>
<td>0.05</td>
<td>0.02</td>
<td>0</td>
</tr>
<tr>
<td>CNS-I-ODS</td>
<td>Bal.</td>
<td>0.1</td>
<td>9.0</td>
<td>0.4</td>
<td>1.2</td>
<td>0.5</td>
<td>0.15</td>
<td>0.25</td>
<td>0.2</td>
<td>0.05</td>
<td>0.02</td>
<td>1.0</td>
</tr>
</tbody>
</table>

Fig. 3 – Elemental mapping images of the A, B and C regions in the rolled CNS-I-ODS.
were hot-forged into sticks with 240 × 240 mm² section as the deformation temperature changed from the initial 1100 °C to the target 900 °C, then hot-rolled into plates with 15 mm thickness followed by water cooling. Finally, the rolled steels were subjected to the heat treatment as following: austenitizing at 1050 °C for 40 min followed by oil quenching, then tempering at 750 °C for 100 min followed by air cooling.

The actual Y content of about 0.698 wt.% for CNS-I-ODS was measured via ICP-AES method. In other words, the yield of Y after VIM & casting for CNS-I-ODS was 69.8%, higher than the literature results, that is, 27.0-56.7% for the Y-bearing CNS-II [23] and 15.4-34.4% for the 0.3C-1.5Si-1.8Mn-0.083Al steel [24]. It has been concluded that the yield of Y during VIM & casting process was dependent on the initial concentration by Qu et al. [24]. Thus the higher yield of Y can be attributed to its excessive addition.

2.2. Characterization

Microstructure examination was conducted on the RD-ND surface of the rolled, quenched and tempered steel. RD is the rolling direction and ND is the normal direction. Samples were mechanically ground, polished, and etched with a solution composed of 5 vol.% HF and 95 vol.% HNO₃. Microstructure was examined by a laser scanning confocal microscope (LSCM, OLYMPUS) and a scanning electron microscope (SEM, LEO 1450) equipped with an energy dispersive spectroscopy (EDS, Oxford Instruments). Besides, second phase particles were also characterized via a transmission electron microscope (TEM, Tecnai F20) equipped with EDS. TEM samples were prepared by mechanical polishing and electrochemical thinning in twin-jet polisher using 10 vol.% perchloric acid and 90 vol.% alcohol solution with a voltage of 30 V at −30 °C.

To determine the phase structure of the Y-bearing particles statistically, electrolytic extraction and X-ray diffraction (XRD) analysis were conducted. Electrolytic extraction was carried out with a solution of 3 g C₆H₅Cl₃N + 10 ml C₂H₅OH + 200 ml CH₃OH at a current density of 100 mA cm⁻² for 6 h. Firstly, the quenched samples were anodically dissolved in the electrolyte solution. Then the residues on the sample surface were scraped off, and the samples were cleaned using an ultrasonic cleaner. Finally, the cleaning solution and the electrolytic solution were collected, and the particles in these solutions were separated by continuous centrifugation and ethanol washing. XRD analysis of the second phases and bulk samples were performed using a Ultima IV diffractometer with Cu Kα radiation of 40 kV and 50 mA. Small angular step of 2θ = 0.02° with a speed of 1° per minute was taken to measure the precise intensity of weak peaks.

Specimens with 5 mm diameter and 25 mm gauge length were subjected to tensile tests using a MTS809 machine with a strain rate of 5.0 × 10⁻⁴ s⁻¹ at RT. Charpy impact tests were conducted on the specimen of 10 × 10 × 55 mm³ with a 45° V-notch at −100 °C-RT. Specimens were kept in liquid nitrogen for 25 min before Charpy impact tests below RT. To obtain the reliable mechanical properties, tensile and Charpy impact tests at each temperature were conducted on three specimens.

3. Results and discussion

3.1. Y-bearing particles

Fig. 1 shows the typical SEM images of the rolled CNS-I and CNS-I-ODS. The CNS-I exhibited uniform martensite structure as shown in Fig. 1(a). By contrast, some banding structures along RD were detected in the CNS-I-ODS as shown in Fig. 1(b). The width of the banding structure region was about 400 µm. Fig. 2 shows the high-magnification SEM image of the banding structure region in the rolled CNS-I-ODS. It can be seen that the banding structure was comprised of second phase particles. Furthermore, the distribution density of these particles was not uniform. Thus the rolled CNS-I-ODS can be divided into A, B and C regions with the decrease of particle distribution density as shown in Fig. 2. To determine the chemical compositions of these particles distributed in the rolled CNS-I-ODS, elemental mapping images of the A, B and C regions were characterized and shown in Fig. 3. Fe and Cr elements were detected in all the three regions. However, Y and O elements were detected only in region A and region B rather than region C. Thus it can be deduced that these particles distributed in A and B regions were comprised of Y and O elements.

To evaluate the evolution of these Y-bearing particles during heat treatment, microstructures of the quenched CNS-I-ODS and tempered CNS-I-ODS were examined and shown in Figs. 4 and 5 and Figs. 6 and 7, respectively. For the quenched CNS-I-ODS, banding structures were also observed and particle distribution was still not uniform. Similarly, three kinds of regions, that is, A, B and C regions were also labelled with the decrease of particle distribution density as shown in Fig. 4. Fig. 5 shows the elemental mapping images of the A, B and C regions for the quenched CNS-I-ODS. Similar to the results of the rolled steel, Fe and Cr elements were found in all the three regions but Y and O elements were detected only in region A and region B rather than region C. Thus it can be concluded
that the banding structures comprised of Y-bearing particles can’t be eliminated via quenching. Fig. 6 shows the SEM image of the tempered CNS-I-ODS. The distribution density of the second particle was still not uniform. Similarly, three kinds of regions were also labelled as A, B and C with the decrease of particle distribution density. And the elemental mapping images of the A, B and C regions for the tempered CNS-I-ODS are shown in Fig. 7. Similar to the results of the rolled and quenched steels, Fe and Cr elements were found in all the three regions while Y and O elements were detected only in region A and region B. Furthermore, Y distribution was consistent with the O distribution in region B, which indicated that these particles distributed along the prior austenite grain boundaries were yttrium oxides. However, Y distribution was inconsistent with the O distribution in region A: the former was well in accordance with the distribution of particles in the grey image while the latter was almost uniform. Similar results were also found in rolled and quenched specimens as
shown in Figs. 3 and 5. Thus it can be inferred that Y-bearing intermetallic compound and yttrium oxides formed in region A simultaneously while yttrium oxides formed in region B for the rolled, quenched and tempered CNS-I-ODS.

To examine the morphology characteristics and chemical compositions of the Y-bearing particles separately, high-magnification SEM image of the tempered CNS-I-ODS is shown in Fig. 8(a). Three kinds of particles including large ellipsoidal ones, moderate spherical ones and small spindle ones were observed. The large ellipsoidal particles with the mean size of 900 ± 400 nm were marked by red arrows, the moderate spherical particles with the mean size of 500 ± 150 nm were marked by blue arrows and the small spindle particles with the mean size of 200 ± 20 nm were marked by yellow arrows. EDS result of the typical large ellipsoidal particle labelled as 1# is shown Fig. 8(b). Except for the matrix elements of Fe, Cr and C, other elements including Y and O were also detected. Moreover, Y content and O content were 12.61 at.% and 44.86 at.%, respectively. Thus it can be deduced that the large ellipsoidal particles are yttrium oxides. EDS result of the typical moderate spherical particle labelled as 2# is shown Fig. 8(c). More Fe, Y and less Cr were detected, which indicated that the moderate spherical particles can be Fe-Y intermetallic compounds. EDS result of the typical small spindle particle labelled as 3# is shown Fig. 8(d). Fe, Cr, C and Ta were the main constituent elements and lots of the small spindle particles were distributed along the prior austenite grain boundaries. Thus it can be inferred that the small spindle particles are M23C6. In brief, 900 nm yttrium oxides and 500 nm Fe-Y intermetallic compounds as well as 200 nm M23C6 are the dominate second phase particles in the tempered CNS-I-ODS. In addition, the relationship between yttrium oxides and Fe-Y intermetallic compounds can be explained via the multi-layer structure presented in Fig. 9. It can be seen that the internal particle labelled as 1# with the diameter of 4.7 μm was surrounded by the external particle labelled as 2# with the diameter of about 18.8 μm. EDS results of 1# and 2# particles in Fig. 9(a) are shown in Fig. 9(b) and Fig. 9(c), respectively. In Fig. 9(b), except for the matrix elements of Fe, Cr and C, more Y and O elements were detected in the internal particle, which thereby can be confirmed as yttrium oxide. In Fig. 9(c), more Fe, Y elements and less Cr, Ta, C elements were observed, which indicated that the external particle can be Fe-Y intermetallic compound. According to the multi-layer structure of yttrium oxide and Fe-Y intermetallic compound, it can be inferred that yttrium oxide formed earlier than Fe-Y intermetallic compound in the molten steel. Then as a non-spontaneous nucleation core, the yttrium oxide was coated by the Fe-Y intermetallic compound. In order to characterize the small Y-bearing particles, TEM analysis was performed on the quenched CNS-I-ODS to minimize the noise from carbides. Fig. 10(a) shows the TEM image of the quenched CNS-I-ODS. Lots of particles were distributed uniformly in the matrix. Elemental mapping images of the marked region in Fig. 10(a) are shown in Fig. 10(b). It can be seen that the main constituent elements were Y, W, Ta and almost no Fe, Cr, O were detected, which indicated that the small particles were Y-W-Ta intermetallic compounds. Size distribution of the particles in Fig. 10(a) is shown in Fig. 10(c), which demonstrated that most of the Y-W-Ta intermetallic compounds exhibited the size ranging from 15 nm to 30 nm and the mean size was about 26 nm.

To determine the phase structure of the Y-bearing particles statistically, electrolytic extraction was conducted on the quenched CNS-I-ODS and the SEM image of the extracted residue is shown in Fig. 11(a). It can be seen that lots of small particles with the size of 300–800 nm were distributed on the large particle surface. EDS results of the typical small particle, that is, 1# and large particle, that is, 2# are shown in Fig. 11(b) and Fig. 11(c), respectively. Cl, C, N, O elements were detected in both particles simultaneously and Y element was only found in the 1# particle. Furthermore, 1# particle displayed more O element compared to the 2# particle. Thus it can be deduced that the large particle is derived from the electrolyte and the small particle is yttrium oxide. Subsequently, XRD analysis was carried out on the extracted residues and the result is shown in Fig. 11(d). High intensity of Cr2H5O3N•HCl as given in JCPDS file (53-1468) can be resulted from the electrolyte comprised of CrH12ClN, CrH5O3N and CH3OH. Besides, some peaks with low intensity were well accordance with the standard characteristics of monoclinic Y2O3 as given in JCPDS file (44-0399). And the size distribution of Y2O3 in Fig. 11(a) is shown in Fig. 11(e). Most of the Y2O3 particles exhibited the size ranging from 200 nm to 800 nm and the mean size was about 550 nm. The cumulative results of SEM, EDS and XRD of the extracted residues revealed that Y2O3 particles have been introduced into the F/M matrix successfully. Besides, XRD analysis was also conducted on the bulk samples including quenched CNS-I, hot rolled CNS-I-ODS, quenched CNS-I-ODS, tempered CNS-I-ODS and the results are shown in Fig. 12. For the quenched CNS-I, only (Fe, Cr) matrix was detected. For the rolled, quenched and tempered CNS-I-ODS, Y(O, C) phase whose peaks are very close to Y2O3C0.3 as given in JCPDS file (38–1114) was also detected except for the (Fe, Cr) matrix. However, peaks of Fe-Y and Y-W-Ta intermetallic compounds were not observed due to their low contents.
Summarily, two kinds of Y-bearing particles were formed in the CNS-1-ODS matrix prepared via VIM & casting technique. One was the yttrium oxides including $\text{Y}_2\text{O}_3$ (see Fig. 11) and Y(O, C) phase (see Fig. 12). The other was Y-bearing intermetallic compounds including Fe-Y phase (see Fig. 9) and Y-W-Ta phase (see Fig. 10). Yttrium oxide displayed larger mean size of 550–900 nm compared to 26–500 nm for Y-bearing intermetallic compounds due to the following reasons: On the one hand, the solid metallic Y would be dissolved into the molten steel at 1600 °C during VIM [26]. Then the dissolved Y would react with activated O immediately as the Gibbs free energy of $\text{Y}_2\text{O}_3$ in liquid steel is about $-280.583 \text{kJ} \cdot \text{mol}^{-1}$ at 1600 °C [27]. By contrast, the typical $\text{Fe}_2\text{Y}_2$ intermetallic compound would be generated until the liquid steel temperature decreased to 1400 °C [26]. On the other hand, $\text{Y}_2\text{O}_3$ exhibits high interface energy and poor wettability with liquid steel, which will further aggravate its coarsening and agglomeration [9].

Fig. 7 – Elemental mapping images of the A, B, C regions in the tempered CNS-1-ODS.
3.2. Matrix microstructure

Fig. 13 shows the LSCM and SEM images of the tempered CNS-I and CNS-I-ODS. The dominate microstructure of the CNS-I was fully tempered martensite due to the martensite transformation caused by oil quenching. Compared to the CNS-I, δ-ferrite and martensite were the main structure in the tempered CNS-I-ODS as Y is the ferrite-favoring element [23]. And the area fraction of the δ-ferrite phase was about 5.4%. The average prior austenite grain size was about 14.4 and 11.7 μm for the tempered CNS-I and CNS-I-ODS, respectively. Grain refinement in the CNS-I-ODS can be resulted from the facilitation of grain boundary nucleation and inhibition of grain boundary movement induced by Y-bearing particles.

The addition of Y not only influenced the average prior austenite grain size but also effected the carbide precipitation. The comparison between Fig. 13(c) and (d) demonstrated that more carbides were precipitated in the tempered CNS-I-ODS compared to the CNS-I. Furthermore, carbides were distributed uniformly in the tempered martensite matrix for the CNS-I while preferentially along the grain boundaries of δ-ferrite for the CNS-I-ODS and the carbides along δ-ferrite boundaries exhibited larger size. Briefly, Y addition resulted in the coarsening of carbides, especially for the carbides along δ-ferrite boundaries in the tempered CNS-I-ODS. Distribution and size characteristics of the carbides in the tempered CNS-I-ODS are determined by the redistribution of carbon during austenitizing. The dissolved carbon content in martensite can be estimated using a mass balance calculation and expressed in Eq. (1) [28] as following:

$$C_M = \frac{C_{total} - C_f V_f}{1 - V_f}$$

where $C_{total}$ is the total carbon content in matrix, $C_f$ is the carbon content in ferrite, $C_M$ is the carbon content in martensite,
3.3. Mechanical properties

Fig. 14 shows the engineering stress-engineering strain curves of the tempered CNS-I and CNS-I-ODS. Ultimate tensile strength (UTS), yield strength (YS) and elongation of the CNS-I were about 660 ± 4.6 MPa, 518 ± 7.7 MPa and 23.5 ± 1.1%. By contrast, the UTS, YS and elongation of the CNS-I-ODS were about 622 ± 4.2 MPa, 482 ± 4.7 MPa and 22.7 ± 0.6%. Obviously, CNS-I-ODS exhibited similar elongation and lower strength compared to the CNS-I, which can be attributed to the large yttrium oxides. In the conventional ODS alloys, microcracks are easier generated at the interface between matrix and large second phase due to the lattice mismatch [29–31], which would produce higher stress concentration at the tips of those microcracks and further result in the crack propagation and sample failure. Similarly, the interface of large yttrium oxides/matrix could be also the preferential sites for crack nucleation and finally reduced the strength of the CNS-I-ODS [32].

Fig. 15 shows the Charpy impact curves of the tempered CNS-I and CNS-I-ODS. The absorbed energy values of CNS-I were higher than those of CNS-I-ODS at each tested temperature. Furthermore, ductile-brittle transition temperature (DBTT) was about −50 and −42 °C for the CNS-I and CNS-I-ODS, respectively. The Charpy impact performance was well accordance with the tensile results for both steels: similar
Fig. 11 – SEM image of the extracted residues from the quenched CNS-I-ODS (a). EDS spectrums of the marked 1# (b) and 2# (c) particles. XRD spectrum of the extracted residues (d). Size distribution of the Y-bearing particle (e).

Fig. 12 – XRD spectrums of the quenched CNS-I, rolled CNS-I-ODS, quenched CNS-I-ODS and tempered CNS-I-ODS.

elongation and lower strength of the CNS-I-ODS led to the lower absorbed energy and higher DBTT, which can be also attributed to the large yttrium oxides in essence. However, it is acceptable for the strength difference of about 38 MPa and DBTT difference of about 8°C between CNS-I and CNS-I-ODS although some large yttrium oxides were distributed in the latter steel. In the future, Y content will be optimized to refine the Y-bearing particles, especially for the yttrium oxides.
4. Conclusions

In the present study, we tried to fabricate the CNS-I-ODS by adding Y$_M$ into the molten steel via VIM & casting method. LSCM, SEM-EDS, TEM-EDS, XRD characterizations were conducted to determine the distribution characteristics, chemical compositions, crystal structure and size distribution of the Y-bearing particles. Besides, the effect of Y addition on matrix microstructure and mechanical properties was evaluated. The
principal conclusions derived from the experimental work are as follows:

1) Some banding structures comprised of yttrium oxides and Y-bearing intermetallic compounds formed along rolling direction.

2) Two kinds of Y-bearing particles were formed in the CNS-I-ODS matrix. One was the yttrium oxides including Y2O3 and Y(O, O) phases. The other was Y-bearing intermetallic compounds including Fe-Y phase and Y-W-Ta phase. Yttrium oxide displayed larger mean size of 550–900 nm compared to 26–500 nm for Y-bearing intermetallic compounds.

3) As the ferrite-favoring element, Y facilitated the formation of δ-ferrite accompanied by martensite matrix and further promoted the precipitation of carbides. Besides, the Y-bearing particles led to the refinement of prior austenite grains.

4) It is acceptable that the CNS-I-ODS exhibited similar elongation of 22.7–23.5%, slightly lower strength of 622 MPa and higher ductile-brittle transition temperature of −42 °C compared to the Y-free steel, although some large yttrium oxides were distributed in the former matrix.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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