

# Effect of yttria inclusion on room temperature tensile properties of investment cast TiAl

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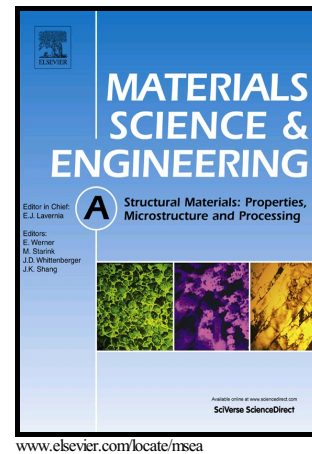
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## Effect of yttria inclusion on room temperature tensile properties of investment cast TiAl

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### Abstract

Yttria is the best face coat material in the shell mold for the investment casting of  $\gamma$ -TiAl alloys. However, yttria inclusions may occur in the cast products and there is a lack of knowledge about the influence of yttria inclusions on the mechanical properties of TiAl. In the present study, the effect of yttria inclusions on room temperature tensile properties of two investment cast TiAl alloys, Ti-45Al-2Mn-2Nb (at.%) + 0.08 vol.% TiB<sub>2</sub> and Ti-46 Al-2 Nb-2Cr-0.15B (at.%), were studied. The results show that tensile failure tends to initiate from yttria inclusions, resulting in reduced plastic strain and ultimate strength. Yttria inclusions differ in location (surface / internal), size, and morphology (agglomerate / whole piece), those with larger sizes or located at the sample surface being more harmful. The tensile properties of inclusion containing TiAl parts can be estimated using the size and location determined by X-ray inspection. The critical size at which inclusions become detrimental to the mechanical properties of TiAl was discussed.

Keywords: TiAl, investment casting, yttria inclusion, tensile properties

### 1. Introduction

TiAl based alloys are light-weight structural materials with excellent high-temperature performance. Due to reduced component weight and increased fuel efficiency, TiAl based alloys show great potential in fields such as the aerospace and automobile industries [1, 2]. Components including low pressure turbine (LPT) blades, turbine wheels [3] and exhaust valves [4] are now being produced with TiAl alloys.

Investment casting is the most cost-effective method of producing near-net-shape TiAl components [5, 6], and ceramic shell mold production is one of the key steps in the casting route [7]. As TiAl melts are highly reactive, the face coat of the mold is required to be highly inert. Much research has been done on face coat materials and

yttria turns out to be the best option [8-12]. Sintering of yttria is very demanding, and at least 1700°C is needed in order to get a relative high density [13]. However, high sintering temperature will raise cost and, more importantly, the mold will be too hard to deform in the cooling process subsequent to casting, which may cause cracking of the cast products. Therefore an intermediate sintering temperature (1000~1400°C) is usually preferred. Nevertheless, this will result in weaker bonding between yttria particles, and the loose particles may be washed off the mold, swept into the molten metal and become yttria inclusions in the cast products [12, 14]. The detrimental effects of nonmetallic inclusions on mechanical properties of steels and superalloys have been extensively reported [15, 16], while similar effects on TiAl remain unknown. In addition, ductility of TiAl at room temperature is poor and has low repeatability, which restrict its more extensive application [17]. Therefore evaluating the effect of yttria inclusions on the mechanical properties of TiAl is of much importance. In this work, test bars of two typical TiAl based alloys for manufacturing low pressure turbine blades (i.e. 45XD by Rolls-Royce and 4622 based on 4822 by GE) were produced by investment casting using yttria face-coated molds, and room temperature tensile properties of the inclusion containing alloys were investigated; the effects of yttria inclusion size, location and morphology were discussed.

## 2. Materials and experiments

### 2.1 Materials and processing

The TiAl alloys used in this study were two typical alloys, Ti-45Al-2Mn-2Nb (at.%) + 0.08 vol.% TiB<sub>2</sub> (45XD) and Ti-46Al-2Nb-2Cr-0.15B (at.%) (4622). Ingots produced with twice VAR (vacuum arc melting) were remelted by ISM (induction skull melting) and centrifugal cast into a mold with an yttria face coat, producing test bars 16 mm in diameter and 210 mm in length. Table 1 shows chemical compositions of the cast rods. The cast rods were hot isostatic pressed (HIPed) at 1260°C and 150 MPa for 4 hours. Heat-treatment was conducted at 1000°C for 4 hours followed by furnace cooling.

### 2.2 Tensile test

The HIPed and heat treated alloys were machined into tensile samples with a gauge diameter of 5 mm and a gauge length of 25 mm. 50 samples of 45XD and 30 samples of 4622 were tested. All the samples were electropolished in order to eliminate machining marks.

Tensile tests were conducted at room temperature. Plastic strain was measured with an extensometer at a strain rate of  $3 \times 10^{-4} \text{ s}^{-1}$  before yielding and  $1.6 \times 10^{-3} \text{ s}^{-1}$  after yielding.

### 2.3 Microstructure studies

The metallographic structures of the heat treated alloys were examined using optical microscopy. The samples were etched by modified Kroll's reagent (5% HF, 10% HNO<sub>3</sub> and 85% H<sub>2</sub>O).

Before tensile testing, traditional 2D X-ray imaging with a resolution of about 100  $\mu\text{m}$  was conducted on all the samples as a preliminary detection method for inclusions. Then two samples, one with inclusions detected and one without, were subjected to 3D high resolution X-ray tomography (XRT) on an Xradia VersaXRM-500 3D X-Ray microscope, the detection range being 5 mm in length and 5 mm in diameter from the gauge section of the tensile samples.

After tensile testing, the fracture surfaces were examined on a Shimadzu SSX-550 scanning electron microscope (SEM). Methods including secondary electron imaging (SE), back scattered electron imaging (BSE) and energy dispersive spectroscopy (EDS) were used for inclusion characterization.

### 3. Results

Microstructures of the two alloys after heat treatment are shown in Fig. 1. 45XD has a fully lamellar microstructure with an average grain (lamellar colony) size of about 70  $\mu\text{m}$ , and few equiaxed gamma grains were observed. Microstructure of 4622 is nearly lamellar, the grain size is similar to 45XD, but contains some equiaxed gamma grains at grain boundaries.

#### 3.1 3D high resolution X-ray tomography

3D high resolution X-ray tomography results are shown in Fig. 2. In addition to the large inclusion detected with traditional X-ray inspection (which caused tensile failure in the subsequent tensile test), more inclusions were observed and most of them are smaller than 60  $\mu\text{m}$  (equivalent spherical diameter).

#### 3.2 Tensile properties

Tensile test results of all the 45XD and 4622 samples are summarized in Table 2. 3 samples exhibited brittle fracture (plastic strain < 0.2%), with no yield strength obtained. For 45XD, yield strength is between 509 MPa and 544 MPa, ultimate strength is between 505 MPa and 681 MPa and plastic strain is between 0.10% and 1.71%. For 4622, yield strength is between 477 MPa and 508 MPa, ultimate strength is between 464 MPa and 601 MPa and plastic strain is between 0.08% and 1.56%. For both alloys, ultimate strength and plastic strain are more scattered than yield strength, and typical strain-stress curves (Fig.3) show that the stress-strain behavior of the samples with varying tensile properties are similar before fracture.

#### 3.3 Fracture surface observation

Both 45XD and 4622 exhibit mostly translamellar fracture and their fracture surfaces are similar. Fig. 4 shows typical fracture surfaces. For samples with good tensile properties (higher ultimate strength and plastic strain, Fig. 4a), no defect was observed on the fracture surfaces. For samples with relatively poor tensile properties (lower ultimate strength and plastic strain), however, cracks were found to initiate from yttria inclusions (Fig. 4b, 4c, 4d) or in some cases from lamellae that are oriented

nearly perpendicular to the loading axis (Fig. 4e, 4f).

## 4. Discussion

### 4.1 Characterization of yttria inclusions and their effects on tensile properties

Samples failed from yttria inclusions usually exhibited inferior tensile properties (lower ultimate strength and plastic strain). The crack initiating yttria inclusions differ in size, location and morphology (Fig. 5). Fig. 6 shows the tensile properties of the alloys containing inclusions of different location and morphology plotted against inclusion size. For comparison, tensile properties of samples without yttria inclusion observed on fracture surface are also listed (samples which failed from facets are not included). Different inclusions have different effect on tensile properties. Inclusions located at the sample surface are evidently more harmful than the internal ones. Inclusion size is also important, with plastic strain and ultimate strength decreasing with increasing inclusion size. The influence of inclusion shape is uncertain since there are only 2 samples with whole piece inclusions and their tensile properties are close to samples with agglomerate inclusions of similar location and size. Data in Fig. 6 is a helpful prediction of the tensile properties of inclusion containing TiAl parts, as the inclusion size and location can be determined with non-destructive X-ray inspection.

Mismatch of thermal expansion coefficients and Young's moduli between inclusion (yttria particles) and matrix (TiAl) leads to a tensile stress around inclusions [18] as well as uncoordinated deformation under loading. Strain will concentrate either inside the inclusion or at the matrix/ inclusion interface, resulting in crack initiation inside the inclusion (yttria particles were observed on both halves of the sample, confirming this cracking of yttria inclusions). Tensile failure in titanium and TiAl alloys have been found to initiate from cracked boride participates [19],  $\alpha$  particles [20] and lamellae with specific orientation (Fig. 4e, 4f). In these cases, cracks can initiate before plastic strain reaches 0.2% [21]. Due to the low toughness of TiAl, cracks will propagate rapidly when load reaches the critical value. Therefore, tensile properties of TiAl are strongly influenced by these cracks. The detrimental effect of a crack can be quantified by the stress concentration it generated. The maximum stress intensity factor caused by a crack,  $K_{I_{max}}$ , is given approximately by [22]:

$$K_{I_{max}} = 0.5\sigma_0\sqrt{\pi\sqrt{area}} \quad (1)$$

for an internal inclusion, and by:

$$K_{I_{max}} = 0.65\sigma_0\sqrt{\pi\sqrt{area}} \quad (2)$$

for a surface inclusion. Here  $\sigma_0$  is the tensile stress and 'area' is the area of the inclusion on fracture surface. As shown in equations (1) and (2), the  $K_{I_{max}}$  caused by surface and larger inclusions are higher than those of internal and smaller inclusions, which explains why larger and surface inclusions lead to worse tensile properties.

## 4.2 Critical inclusion size

The crack initiating inclusions observed are usually hundreds of microns in size and the smallest one is 70  $\mu\text{m}$  (converted to equivalent diameter from square root of area, Fig. 2). However, X-ray tomography results suggest that most of the inclusions are smaller than 60  $\mu\text{m}$  (Fig. 2), and these small inclusions were also found in the samples that did not fail from inclusions (such as 4622-11). This indicates the existence of a critical size below which yttria inclusions do not influence the tensile properties of TiAl. The smallest crack initiating inclusion size, 70  $\mu\text{m}$ , is close to the grain size. The work of Nazmy et al. obtained similar results: surface cracks smaller than lamellar grain size have no influence on high cycle fatigue life of TiAl [23]. Moreover, the rogue grains or 'facets' mentioned previously are also larger than the average grain size. These results suggest that a brittle object, whether it is a nonmetallic inclusion, a boride particle, a notch, a cluster of small grains or one single grain with specific lamellar orientation, is harmful to the mechanical properties of TiAl only when it is larger than the grain size. Therefore control of inclusion should be focused on decreasing the size of the largest inclusion and this can be achieved through optimizing mold making technology.

Property consistency and predictability is crucial for safety-critical parts such as turbine blades. As yttria inclusions are inevitable in investment cast TiAl parts, establishment of inclusion-related quality specifications is indispensable and the results in this research provide a reference.

## 5. Conclusion

Yttria inclusions introduced into TiAl parts during the investment casting process tend to act as crack initiation sites and are detrimental to the room temperature tensile properties of the parts, especially ultimate strength and plastic strain. The effects of yttria inclusions are mainly determined by inclusion location and size. Those located at the sample surface or with larger sizes are more detrimental as they cause a higher stress intensity. The room temperature tensile properties of TiAl can be estimated with inclusion size and location determined by X-ray tomography. The critical inclusion size to cause tensile failure is close to the grain size.

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Table 1 Chemical composition of the investigated alloys.

| Alloy    | Composition | Ti   | Al    | Nb   | Mn   | Cr   | B    | H      | N      | O      |
|----------|-------------|------|-------|------|------|------|------|--------|--------|--------|
| 45X<br>D | wt. %       | Bal. | 30.80 | 4.80 | 2.85 | /    | 0.25 | 0.0023 | 0.0025 | 0.0640 |
|          | at. %       | Bal. | 44.70 | 2.02 | 2.03 | /    | 0.91 | 0.0894 | 0.0070 | 0.1566 |
| 4622     | wt. %       | Bal. | 32.00 | 4.90 | /    | 2.65 | 0.05 | 0.0020 | 0.0045 | 0.0720 |
|          | at. %       | Bal. | 46.30 | 2.06 | /    | 1.99 | 0.18 | 0.0775 | 0.0125 | 0.1757 |

Table 2. Tensile properties of 45XD and 4622 at room temperature.

| Alloy | 45XD                       |                         |       |     |                            |                         |       | Alloy | 4622                       |                         |       |
|-------|----------------------------|-------------------------|-------|-----|----------------------------|-------------------------|-------|-------|----------------------------|-------------------------|-------|
| No.   | Tensile properties         |                         |       | No. | Tensile properties         |                         |       | No.   | Tensile properties         |                         |       |
|       | R <sub>p0.2</sub><br>(MPa) | R <sub>m</sub><br>(MPa) | A (%) |     | R <sub>p0.2</sub><br>(MPa) | R <sub>m</sub><br>(MPa) | A (%) |       | R <sub>p0.2</sub><br>(MPa) | R <sub>m</sub><br>(MPa) | A (%) |
| 1     | 539                        | 611                     | 0.71  | 31  | 541                        | 610                     | 0.68  | 1     | 493                        | 509                     | 0.33  |
| 2     | 523                        | 591                     | 0.74  | 32  | 534                        | 659                     | 1.39  | 2     | 494                        | 540                     | 0.59  |
| 3     | -                          | 505                     | 0.10  | 33  | 524                        | 659                     | 1.52  | 3     | 483                        | 589                     | 1.50  |
| 4     | 515                        | 646                     | 1.71  | 34  | 541                        | 681                     | 1.67  | 4     | 492                        | 511                     | 0.35  |
| 5     | 527                        | 647                     | 1.33  | 35  | 535                        | 672                     | 1.68  | 5     | 493                        | 551                     | 0.74  |
| 6     | 532                        | 577                     | 0.40  | 36  | 536                        | 657                     | 1.47  | 6     | 494                        | 601                     | 1.56  |
| 7     | 524                        | 638                     | 1.31  | 37  | 504                        | 624                     | 1.38  | 7     | -                          | 496                     | 0.18  |
| 8     | 520                        | 633                     | 1.26  | 38  | 535                        | 647                     | 1.16  | 8     | 490                        | 551                     | 0.76  |
| 9     | 544                        | 648                     | 0.97  | 39  | 533                        | 655                     | 1.42  | 9     | 487                        | 576                     | 1.15  |
| 10    | 520                        | 601                     | 0.80  | 40  | 544                        | 675                     | 1.56  | 10    | 475                        | 523                     | 0.68  |
| 11    | 523                        | 597                     | 0.66  | 41  | 539                        | 616                     | 0.68  | 11    | 489                        | 589                     | 1.37  |
| 12    | 534                        | 640                     | 1.13  | 42  | 535                        | 652                     | 1.26  | 12    | 486                        | 560                     | 0.95  |
| 13    | 532                        | 641                     | 1.11  | 43  | 537                        | 638                     | 1.04  | 13    | 508                        | 600                     | 1.28  |
| 14    | 525                        | 648                     | 1.35  | 44  | 520                        | 615                     | 0.87  | 14    | 492                        | 533                     | 0.5   |
| 15    | 543                        | 658                     | 1.29  | 45  | 528                        | 639                     | 1.22  | 15    | 493                        | 521                     | 0.39  |
| 16    | 544                        | 572                     | 0.37  | 46  | 520                        | 612                     | 0.89  | 16    | 494                        | 580                     | 1.15  |
| 17    | 517                        | 537                     | 0.31  | 47  | 537                        | 665                     | 1.37  | 17    | 481                        | 591                     | 1.49  |
| 18    | 520                        | 576                     | 0.50  | 48  | 535                        | 648                     | 1.08  | 18    | 477                        | 564                     | 1.11  |
| 19    | 509                        | 567                     | 0.50  | 49  | 523                        | 594                     | 0.62  | 19    | 487                        | 589                     | 1.34  |
| 20    | 527                        | 673                     | 1.61  | 50  | 523                        | 600                     | 0.66  | 20    | -                          | 464                     | 0.08  |
| 21    | 530                        | 666                     | 1.62  |     |                            |                         |       | 21    | 486                        | 538                     | 0.58  |
| 22    | 519                        | 656                     | 1.44  |     |                            |                         |       | 22    | 488                        | 537                     | 0.58  |
| 23    | 525                        | 666                     | 1.55  |     |                            |                         |       | 23    | 487                        | 535                     | 0.56  |
| 24    | 523                        | 656                     | 1.59  |     |                            |                         |       | 24    | 482                        | 582                     | 1.33  |
| 25    | 522                        | 643                     | 1.35  |     |                            |                         |       | 25    | 487                        | 526                     | 0.43  |
| 26    | 516                        | 653                     | 1.61  |     |                            |                         |       | 26    | 479                        | 551                     | 0.79  |
| 27    | 520                        | 647                     | 1.40  |     |                            |                         |       | 27    | 487                        | 572                     | 0.98  |
| 28    | 522                        | 643                     | 1.22  |     |                            |                         |       | 28    | 488                        | 575                     | 1.09  |
| 29    | 518                        | 651                     | 1.39  |     |                            |                         |       | 29    | 490                        | 577                     | 1.11  |
| 30    | 535                        | 652                     | 1.31  |     |                            |                         |       | 30    | 479                        | 482                     | 0.22  |

R<sub>p0.2</sub>: yield strength; R<sub>m</sub>: ultimate strength; A: plastic strain.

Figure Captions:

Fig. 1. Microstructures of (a) 45XD and (b) 4622.

Fig. 2. 3D high resolution X-ray tomography results: (a) is the schematic diagram of the detection range, (b) shows inclusions observed in sample 4622-10 and (c) is the size distribution of inclusions in samples 4622-10 and 4622-11.

Fig. 3. Typical stress-strain curves of (a) 45XD and (b) 4622 with varying tensile properties.

Fig. 4. Fracture surfaces of 45XD. (a) Sample with no defects observed (sample 45XD-7:  $R_{p0.2} = 524$  MPa,  $R_m = 638$  MPa,  $A = 1.31\%$ ); (b) sample failed from an yttria inclusion (sample 45XD-18:  $R_{p0.2} = 520$  MPa,  $R_m = 576$  MPa,  $A = 0.50\%$ ), (c) being the magnified BSE image and (d) the EDS result of the inclusion; (e) sample failed from a 'facet' (lamella perpendicular to the loading axis), (f) being an enlarged image of the facet (sample 45XD-9:  $R_{p0.2} = 544$  MPa,  $R_m = 648$  MPa,  $A = 0.97\%$ ).

Fig. 5. Categorization of yttria inclusions according to location and morphology: (a) surface/ agglomerate, (b) internal/ agglomerate, (c) surface/ whole piece, (d) internal/ whole piece. An agglomerate inclusion consists of segmented small yttria particles, and a whole piece inclusion consists of one big yttria particle.

Fig. 6. Tensile properties of 45XD (a, c, e) and 4622 (b, d, f) with different inclusion location and morphology against size.

Fig. 7. Fracture surfaces of a tensile test sample, with yttria inclusions observed on both halves of the fractured sample.

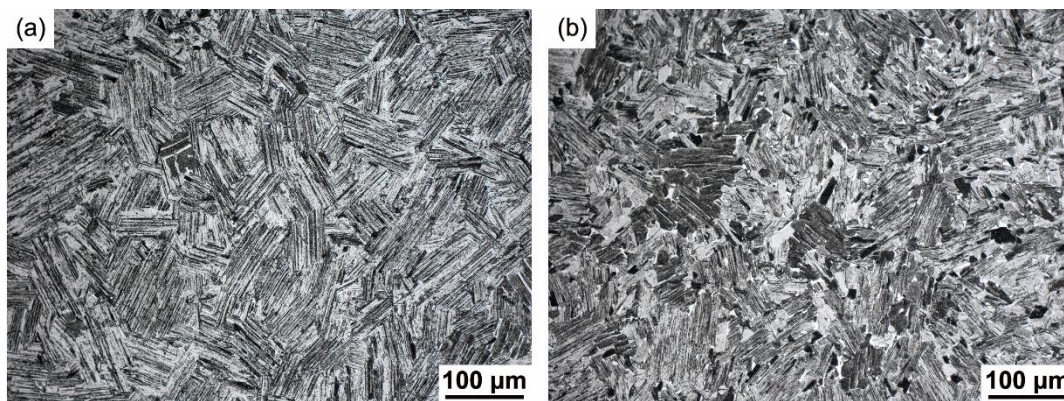


Fig. 1. Microstructures of (a) 45XD and (b) 4622.

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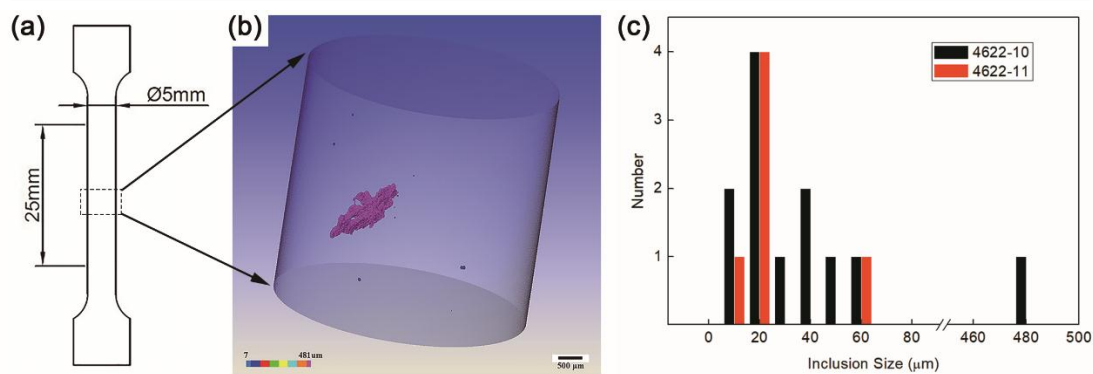


Fig. 2. 3D high resolution X-ray tomography results: (a) is the schematic diagram of the detection range, (b) shows inclusions observed in sample 4622-10 and (c) is the size distribution of inclusions in samples 4622-10 and 4622-11.

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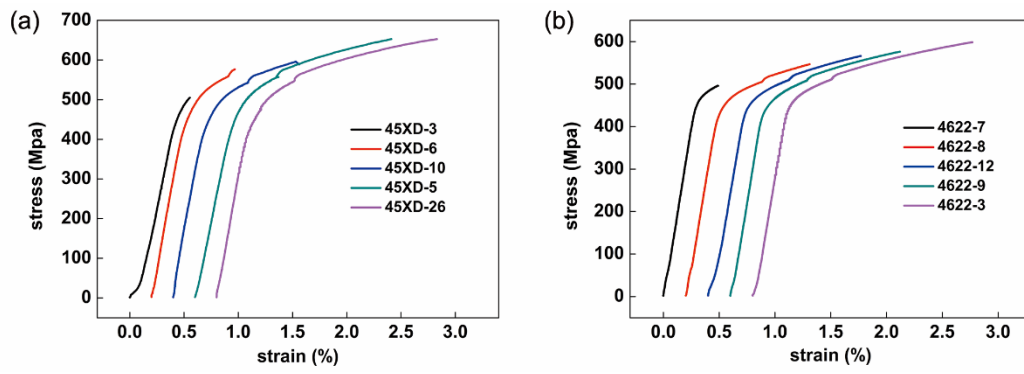


Fig. 3. Typical stress-strain curves of (a) 45XD and (b) 4622 with varying tensile properties.

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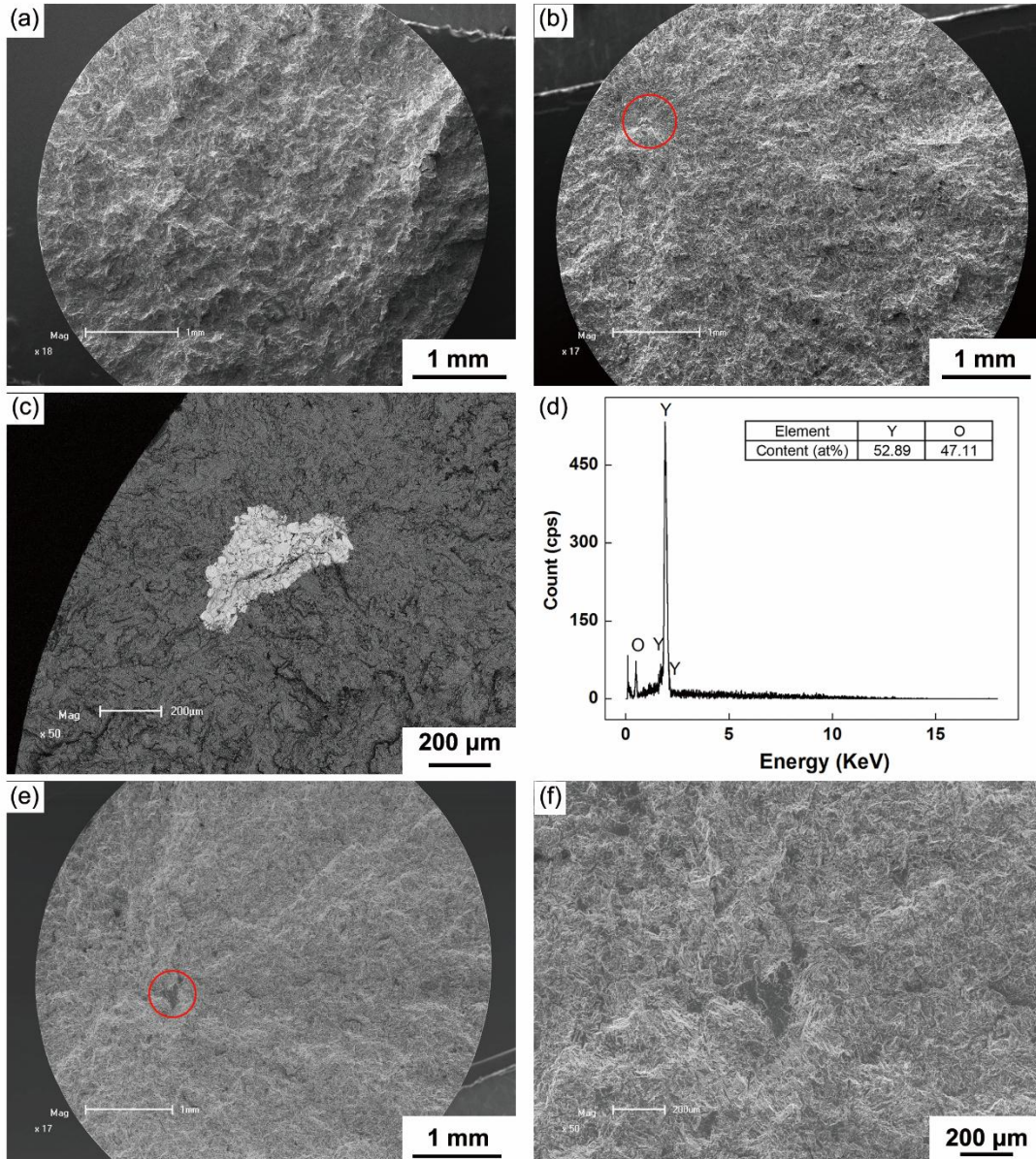


Fig. 4. Fracture surfaces of 45XD. (a) Sample with no defects observed (sample 45XD-7:  $R_{p0.2} = 524$  MPa,  $R_m = 638$  MPa,  $A = 1.31\%$ ); (b) sample failed from an yttria inclusion (sample 45XD-18:  $R_{p0.2} = 520$  MPa,  $R_m = 576$  MPa,  $A = 0.50\%$ ), (c) being the magnified BSE image and (d) the EDS result of the inclusion; (e) sample failed from a 'facet' (lamella perpendicular to the loading axis), (f) being an enlarged image of the facet (sample 45XD-9:  $R_{p0.2} = 544$  MPa,  $R_m = 648$  MPa,  $A = 0.97\%$ ).



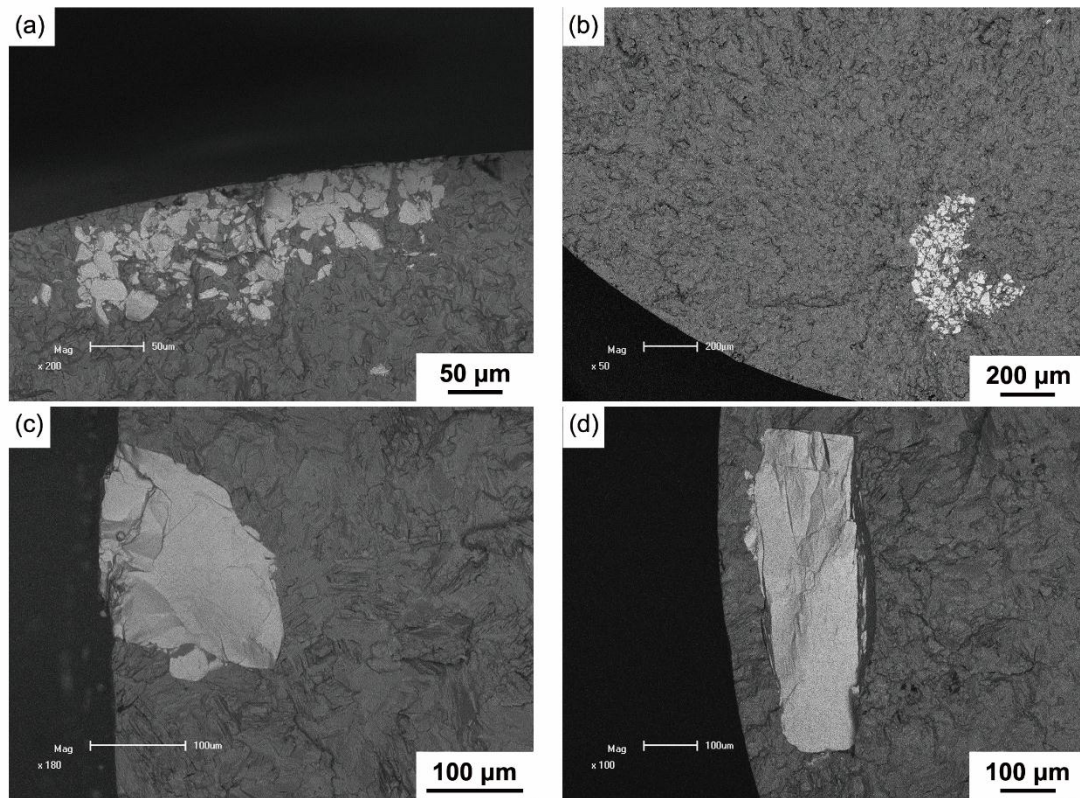


Fig. 5. Categorization of yttria inclusions according to location and morphology: (a) surface/ agglomerate, (b) internal/ agglomerate, (c) surface/ whole piece, (d) internal/ whole piece. An agglomerate inclusion consists of segmented small yttria particles, and a whole piece inclusion consists of one big yttria particle.

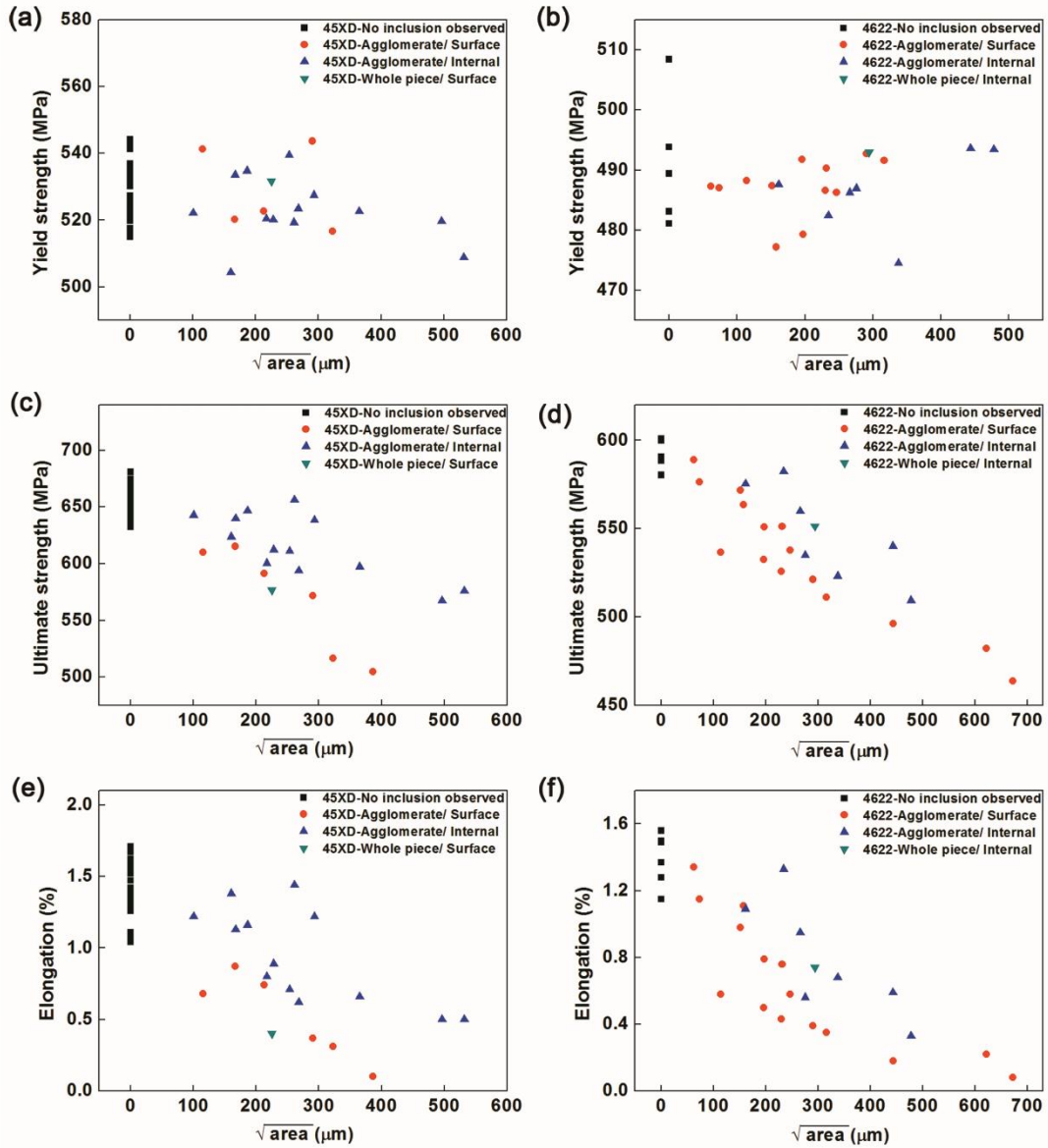


Fig. 6. Tensile properties of 45XD (a, c, e) and 4622 (b, d, f) with different inclusion location and morphology against size.

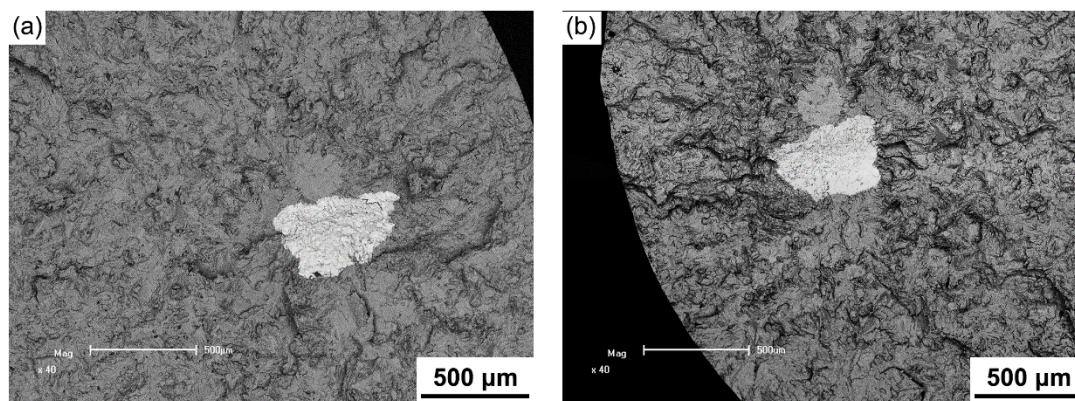


Fig. 7. Fracture surfaces of a tensile test sample, with yttria inclusions observed on both halves of the fractured sample.

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