Effect of Rare Earth Metals on the Microstructure and Impact Toughness of a Cast 0.4C–5Cr–1.2Mo–1.0V Steel

Jie LAN, Junjie HE, Wenjiang DING, Qudong WANG and Yanping ZHU

School of Materials Science and Engineering, Shanghai Jiao Tong University, P.R.China. E-mail: lj1325@263.net

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Die-making technology directly produced by precise casting technology such as investment and ceramic casting method was a promising technology due to its lower manufacture cost and shorter period in comparison with traditional die-making technology. However, the poor mechanical performance of cast materials, especially low impact toughness, was a main obstacle to the application of cast dies and molds.

With a view to improve the impact toughness of CH13 steel (0.4C–5Cr–1.2Mo–1V steel), which was ever used to cast dies for die casting dies, rare earth was added into CH13 steel in our research. It was shown that the dendrite microstructure in CH13 steel had been ameliorated and the segregation ratios of alloying elements were decreased greatly after the addition of rare earth. Grain boundary carbides in MCH13 (CH13+ rare earth) steel had also been inhibited. The impact toughness of MCH13 steel had been improved considerably and the fractography of MCH13 displayed more plastic characteristic. Through thermodynamics and two-dimensional lattice disregistry analysis, it was demonstrated that rare earth inclusions in MCH13 steel act as the heterogeneous nuclei, weaken segregation of alloying elements and improve the impact toughness.

KEY WORDS: cast AISI H13 steel; rare earth; fracture behavior; two-dimensional lattice disregistry; heterogeneous nuclei.
10 g mixed rare earth was plunged into the bottom of down-sprue (2 point in Fig. 1) and a small aluminum disc with another 10 g rare earth was placed at the gate (1 point in Fig. 1) in investment mold. This procedure must be rapid in case of the excessive oxidation of rare earth. The compositions of mixed rare earth are shown in Table 1.

Inductively coupled plasma (ICP) method was used to analyze the chemical compositions of CH13 and MCH13 steels. Four samples from four different positions in MCH13 steel (A, B, C and D point in Fig. 1) were selected in order to examine the homogeneity of rare earth in MCH13 steel. Samples were dissolved in the mixed solution with 1 m/ HF, 3 m/ HNO₃ and 5 m/ HCl under 1.013×10⁶ Pa for 10 min. The chemical compositions of both steels are given in Table 2. It can be seen that Ce and La are distributed evenly in MCH13 steel but Pr and Nd can not be detected owing to their very little amount.

Test samples were annealed at 1 143 K for 120 min in an air furnace, then austenitized at 1 323 K for 30 min in an Ar-controlled furnace and quenched in oil. The air furnace was also used for tempering cycles at 893 K for 120 min twice and cooled in air. The microstructure of the steels was characterized using standard optical metallography. Scanning electron microscopy (PHILPS-SEM515) was used to study the fracture surfaces and inclusions in the steels. Quantitative analysis of inclusions and microcomponents of both steels was undertaken by electron probe microanalysis (JXA-8800RL) and EDAX in SEM. Impact test on unnotched samples of 10×10×55 mm was performed for determining impact toughness with a JB-5 impact test machine.

3. Experimental Results and Discussions

3.1. Cast and Temper Microstructures of CH13 and MCH13 Steels

For the optical microstructure of the samples (Figs. 2(a), 2(b)), the cast microstructure of both steels consists of the light and dark areas. According to the CCT curve of CH13 steel, the dark area is low carbon martensite, pre-solidified structure with lower carbon and alloying elements. The light area is high carbon martensite with some austenite and carbides, which is the structure of higher carbon and alloying elements solidified lately. The light areas in CH13 steel are net-like dispersed showing the obvious dendrite structure while these light areas in MCH13 steel are broken and dot-like dispersed and evident dendrite structure is not observed. Quantitative metallography was carried out by point counting method in order to determine the amount of light areas in both steels. The results, as shown in Table 3, indicate that the addition of RE greatly decreases the amount of light areas in the CH13 steel.

The segregation ratio \( \delta \) of alloying elements was introduced to evaluate the extent of segregation of alloying elements \( \delta = \frac{C_{\text{max}}}{C_{\text{min}}} \), where \( C_{\text{max}} \) is the maximum concentration of \( i \) element at interdendritic area and \( C_{\text{min}} \) is the minimum concentration of \( i \) element at dendritic area. Electron probe microanalysis was used to detect the microcomponents of dendrite and interdendrite areas and over 20 areas were selected (Figs. 2(c), 2(d)). It can be seen from Table 4 that there are no obvious differences in \( \delta_{\text{Si}}, \delta_{\text{Mn}}, \delta_{\text{Cr}}, \delta_{\text{Mo}}, \delta_{\text{V}} \) before and after RE addition. However, \( \delta_{\text{Cr}}, \delta_{\text{Mo}}, \delta_{\text{V}} \) in MCH13 steel were smaller than those in CH13 steel greatly. It can be concluded from the above results that the microstructure of MCH13 steel is more homogeneous than that of the CH13 steel.

It is inevitable that some grain boundary areas of CH13 steel are also interdendritic areas with high concentration of alloying elements due to severe dendrite segregation in CH13 steel. Compared with those areas with low concentration of alloying elements, the curve of carbides in CCT curve of high concentration of alloying elements in CH13 steel will be moved toward left. Therefore, the alloy carbides are inclined to form at those areas with high concentration of alloying elements during the slow cooling rate of annealing, especially at grain boundary with high concentration of alloying elements because grain boundaries can

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**Table 1.** Compositions (mass%) of mixed rare earth.

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<tr>
<th>Element</th>
<th>Ce</th>
<th>La</th>
<th>Pr</th>
<th>Nd</th>
<th>Fe</th>
<th>Si</th>
<th>Mg</th>
<th>Mn</th>
<th>Ca</th>
<th>P</th>
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<td>mass%</td>
<td>50.2</td>
<td>26.67</td>
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<td>0.65</td>
<td>0.01</td>
<td>0.38</td>
<td>0.11</td>
<td>0.01</td>
<td>0.003</td>
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**Table 2.** Compositions (mass%) of CH13 and MCH13 steels.

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<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>S</th>
<th>P</th>
<th>Ce</th>
<th>La</th>
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<td>0.83</td>
<td>0.22</td>
<td>4.99</td>
<td>1.19</td>
<td>0.89</td>
<td>0.012</td>
<td>0.017</td>
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<td>MCH13 A</td>
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<td>0.90</td>
<td>0.22</td>
<td>4.95</td>
<td>1.18</td>
<td>0.88</td>
<td>0.010</td>
<td>0.017</td>
<td>0.0431</td>
<td>0.0104</td>
</tr>
<tr>
<td>MCH13 B</td>
<td>0.39</td>
<td>0.83</td>
<td>0.20</td>
<td>4.95</td>
<td>1.23</td>
<td>0.92</td>
<td>0.012</td>
<td>0.012</td>
<td>0.0433</td>
<td>0.0125</td>
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<tr>
<td>MCH13 C</td>
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<td>0.86</td>
<td>0.23</td>
<td>5.02</td>
<td>1.19</td>
<td>0.92</td>
<td>0.013</td>
<td>0.015</td>
<td>0.0428</td>
<td>0.0090</td>
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<tr>
<td>MCH13 D</td>
<td>0.38</td>
<td>0.89</td>
<td>0.23</td>
<td>4.90</td>
<td>1.22</td>
<td>0.89</td>
<td>0.015</td>
<td>0.013</td>
<td>0.0430</td>
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</table>
act as the nuclei of alloy carbides more easily than other areas. These alloy carbides cannot be eliminated despite high temperature during austenitizing. Compared to the CH13 steel, grain boundary carbides in MCH13 steel have been controlled because of more homogenous cast structure (Figs. 2(e), 2(f)). It was mentioned that these carbides were not formed during quenching owing to the small size of test samples and high cooling rate of quenching.

3.2. Impact Toughness and Fracture Features

3.2.1. Impact Toughness of CH13 and MCH13 Steels at Ambient Temperature

The impact test results were shown in Fig. 3. Each of the values shown in this figure represented the average of at
least ten samples. In contrast to CH13 steel, the impact toughness of MCH13 steel increased from 29 J to 68 J while the same hardness was maintained.

3.2.2. The Fractography of CH13 and MCH13 Steels

The representative fractography of both steels is shown in Figs. 4(a) and 4(b). Both steels display the characteristic of quasi-cleavage (Fig. 4(c)). Compared with CH13 steel, the fracture surface in MCH13 steel is much rougher and possesses more dimples.

Figure 5 is the longitudinal sections of fracture specimens. There are two obvious differences between CH13 and MCH13 steels: firstly, the longitudinal section of MCH13 steel is macroscopic rougher than that of CH13 steel, which is well consistent with the fracture surface of MCH13 steel. Secondly, a great number of secondary cracks adjacent to the fracture surface in MCH13 steel are observed whereas much less in CH13 steel. In CH13 steel, main cracks would propagate and connect together rapidly once the cracks are formed. Secondary cracks near the fracture surface have no time to initiate ahead of fracture. Comparatively, more plastic deformation was generated in MCH13 steel before cracks propagate, especially in and adjacent to the fracture surface with high-stress field. These secondary cracks are formed as the main crack propagates in a catastrophic manner as a consequence of the release of a large amount of stored elastic energy. The little size of the secondary cracks will be an indication of the effectiveness of the microstructural barriers to crack propagation under a decreasing stress intensity. This indicates the MCH13 steel is tougher.

3.2.3. The Fracture Behavior of CH13 and MCH13 Steels

For both steels, cracks propagated mostly transgranularly through the matrix. However, in some areas, the cracks propagated along isolated intergranular networks of carbide in the CH13 steel. According to Smith mode, the network carbides on grain boundaries can act as strong obstacles against motion of partial dislocations. As the results, many partial dislocations pile up and produce a large stress concentration at carbides of grain boundary. This leads to the formation of tunnel-like voids. The cracks along grain boundary can be formed easily by the coalescence of voids. Such microcracks are found at the longitudinal sections of fracture specimens in CH13 steel whereas not in MCH13 steel, which is ascribed to elimination of network carbides impinging on grain boundaries by the addition of RE to CH13 steel (shown in Fig. 6).

Sulphides and oxide inclusions were also an important factor influencing the fracture behavior of CH13 and MCH13 steels. Microcracks initiated at sulphides were found in both in longitudinal sections and fracture surface (Fig. 7), showing that the sulphides can act as the nuclei of quasi-cleavage fracture. Cracked Al₂O₃ was also found at the longitudinal sections of fracture specimens in CH13 steel (as shown in Fig. 8). Although microcrack in Al₂O₃ does not develop across the matrix, this does not adequately deny that the quasi-cleavage can nucleate at oxide inclusions. According to a previous research, the oxide inclusions are larger and more highly elongated than the sulphides. As a result, they can act as more effective local sites of stress concentration.

In MCH13 steel, elongated oxide and sulphide inclusions were replaced by spherical rare earth inclusions. It has been
observed that the cracked rare earth inclusions can also act directly as quasi-cleavage nuclei (Fig. 9). Compared to spherical inclusions, elongated inclusions in steel can give rise to a stress concentration at the tip of inclusions. A crack will be easily created by the stress concentrations and the pile-up of dislocations against the inclusion.\textsuperscript{11)}

Fig. 6. (a) Microcrack initiated at grain boundary carbides in CH13 steel. (b) EDAX energy spectroscopy of alloy carbides. (c) Smith mode.

Fig. 7. Microcrack initiated at sulphide inclusions in CH13 steel. (a) (Fe, Mn)S in the longitudinal section. (b) (Fe, Mn)S in the fracture surface. (c) EDAX energy spectroscopy of sulphides.
3.3. The Mechanism of Heterogeneous Nuclei of RE Additions

The effects of RE additions to steel have been very well known, e.g., prevention of precipitation, coalescence and growth of carbides in martensite, prevention of fracture along grain boundaries of steels containing phosphorus and reduction in temper brittleness of steels,12) and modification of inclusion as observed in our work. Additionally, there is an additional role in cast steel that some kinds of RE inclusions may act as heterogeneous nuclei of cast steel during solidification. Therefore, the behavior of RE inclusions in MCH13 steel was investigated. Because Ce, La and Nd etc. in RE additions have similar chemical behavior and Ce consists mostly, Ce inclusion was investigated and calculated representatively in following section.

3.3.1. Thermodynamics Analysis of Ce₂O₃ during Solidification

Spherical inclusions distributed evenly in MCH13 steel and its linear scanning of elements were shown in Fig. 9. According to the electron probe microanalysis, the compositions of RE inclusion are (%): Ce 49.654, La 13.388, O 28.243, Al 1.629, S 1.659, Fe 5.427, which demonstrated that the inclusion should be Ce₂O₃ (type A of RE oxides).13) Firstly, the calculation of thermodynamics was undertaken in order to investigate whether Ce₂O₃ can form before pre-solidified phase of MCH13 steel.

The equation for formation of Ce₂O₃ is:

\[
\Delta G_{\text{Ce}_2\text{O}_3} = -1888944 + 676.95T + RT \ln \frac{1}{a_{\text{Ce}}^2 \cdot a_{\text{O}}} = -1888944 + 676.95T - 19.15T(2 \log a_{\text{Ce}} + 3 \log a_{\text{O}}), \quad \text{J/mol... (1)}
\]

where \(a\) is the activity, \(T\) is temperature (K), \(a_{\text{Ce}}\) and \(a_{\text{O}}\) are calculated by infinite dilute solution model.
respectively. The values of 

calculated using Eqs. (1) to (4) as follows:

\[ a_i = f_i \% [\text{Ce}] \] ................................................. (2)

\[ \log f_i = e_i^j [\% [\text{Ce}]] + e_i^j [\% [\text{Fe}]] + \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cd • 0.1%, which indicates that Ce<sub>2</sub>O<sub>3</sub> can be formed before the formation of pre-austenite during solidification.

3.3.2. The Calculation of Lattice Disregistry

The boundary energy between the heterogeneous nucleus and the crystallization phase has an effect on nucleus formation and it depends on the structure of the two contacting crystalline faces (arrangement condition, atom size and interatomic spacing of the crystal faces). The value of the two-dimensional lattice misfits usually evaluated where some inclusion may act as the heterogeneous nuclei. The two-dimensional lattice disregistry mathematical model from Bramfitt<sup>15</sup> is:

\[ \delta_{(hkl)_s} = \sum_{i=1}^{3} |\{d_{(uvw)_h} \cos \theta - d_{(uvw)_s}\}| / 3 \times 100\% \] ...................................(6)

where the \((hkl)_h\) is the low index crystal face of the nucleus, the \([uvw]_h\) is the low index crystal orientation in the \((hkl)_h\); the \((hkl)_s\) is the low index crystal face of the new crystal phase, \([uvw]_s\) is the low index crystal orientation in the \((hkl)_s\); \(d_{(uvw)_h}\) and \(d_{(uvw)_s}\) are the interatomic spacing along the \([uvw]_h\) and the \([uvw]_s\), \(\theta\) is the angle between \([uvw]_h\) and \([uvw]_s\).

The relationship of crystal orientation between low index crystal faces of γ-Fe and Ce<sub>2</sub>O<sub>3</sub> is shown in Fig. 11. Results of calculation of two-dimensional lattice disregistry are shown in Table 5. It is demonstrated that the lattice disregistry between the (0001) of Ce<sub>2</sub>O<sub>3</sub> and (100) of is 5.92%, less than 12%<sup>16</sup> which means Ce<sub>2</sub>O<sub>3</sub> can act as the heterogeneous nucleation for initial γ-Fe, ameliorate the segregation of alloying elements and improve impact toughness.

4. Conclusions

The microstructure and impact toughness of CH13 and MCH13 steels were evaluated and compared. The following conclusions can be drawn:

1. The dendrite structure of CH13 steel is refined and the segregation of alloying elements is ameliorated by the addition of rare earth. The structure of MCH13 is more homogeneous than CH13 steel and the impact toughness is increased from 29 J to 68 J.

2. Alloy carbides on grain boundaries are the main factor leading to the fracture of CH13 steel. Elongated oxides and sulphides can also act as the nuclei of quasi-cleavage fracture. In MCH13 steel, elongated oxide and sulphide
inclusions are replaced by spherical rare earth inclusions and cracked rare earth inclusions can act directly as quasi-cleavage nuclei.

(3) By calculation of thermodynamic, it was obtained that the \( \Delta G_{\text{Ce}_2\text{O}_3} \) values at 1 773, 1 823 and 1 873 K are less than zero, which means \( \text{Ce}_2\text{O}_3 \) can form before the pre-solidified phase during solidification.

(4) By analysis of two dimensional lattice disregistry, it was shown that the disregistry between (0001) of \( \text{Ce}_2\text{O}_3 \) and (100) of austenite is only 5.92% less than 12%, which indicates that \( \text{Ce}_2\text{O}_3 \) can effectively act as the heterogeneous nuclei of initial \( \gamma \)-Fe.

### Table 5. Results of calculation of two-dimensional lattice disregistry.

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<tr>
<th>Case</th>
<th>([hkl]_s)</th>
<th>([hkl]_n)</th>
<th>(d[\text{[hkl]}_s, \text{Å})</th>
<th>(d[\text{[hkl]}_n, \text{Å})</th>
<th>(\theta, \text{deg})</th>
<th>(\delta_{[hkl]}^{(\text{LSM})})</th>
</tr>
</thead>
<tbody>
<tr>
<td>((0001)_{\text{Ce}_2\text{O}<em>3}(1\overline{1}0)</em>{\gamma-\text{Fe}})</td>
<td>[1\overline{2}10]</td>
<td>[010]</td>
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<td>3.686</td>
<td>0</td>
<td>5.92</td>
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<tr>
<td></td>
<td>[2\overline{1}10]</td>
<td>[012]</td>
<td>7.872</td>
<td>8.242</td>
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<td>5.92</td>
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<td>[002]</td>
<td>6.817</td>
<td>7.372</td>
<td>0</td>
<td>5.92</td>
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<td>((0001)_{\text{Ce}_2\text{O}<em>3}(1\overline{1}0)</em>{\gamma-\text{Fe}})</td>
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<td>3.686</td>
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