Feasibility research of gaining “refractory high entropy carbides” through in situ carburization of refractory high entropy alloys

Yuanlin Ai
National University of Defense Technology, 495586680@qq.com

Shuxin Bai
National University of Defense Technology

Li’an Zhu
National University of Defense Technology

Yicong Ye
National University of Defense Technology

Follow this and additional works at: http://dc.engconfintl.org/uhtc_iv

Part of the Engineering Commons

Recommended Citation

This Abstract and Presentation is brought to you for free and open access by the Proceedings at ECI Digital Archives. It has been accepted for inclusion in Ultra-High Temperature Ceramics: Materials for Extreme Environment Applications IV by an authorized administrator of ECI Digital Archives. For more information, please contact franco@bepress.com.
Feasibility Research of Gaining “Refractory High Entropy Carbides” Through In Situ Carburization of Refractory High Entropy Alloys

Lian Zhu, Yuanlin Ai, Shuxin Bai
National University of Defense Technology, China
Outline

1. Introduction
2. Experimental
3. Results and discussion
4. Conclusions
1. Introduction

High-entropy alloys (HEAs)
- First proposed by Yeh in 2004
- Excellent comprehensive properties
- 4 core effects

1. Introduction

- High-entropy ultra-high temperature ceramics (HEUHTCs)?
  - Entropy stabilized material?
  - Excellent comprehensive properties?

**High entropy alloy → High entropy carbide ceramic**

HfZrTiTa → (Hf,Zr,Ti,Ta)C
2. Experimental

- Preparation of HfZrTiTa alloy

Hf+Zr+Ta+Ti Powder (1:1:1:1) → Vacuum arc melting → HfZrTiTa HEA
2. Experimental

- **Solid carburization**
  - Pack cementation (900°C, 10h)
  - Carburizing agent (powder):
    - 90%C+10%Na$_2$CO$_3$
    - 90%C+10%CaCO$_3$
    - 100%C
  - Loading amount
    - Full
    - Half full
2. Experimental

**Testing and characterization**

- Micro-hardness
  - Load: 50 g; Holding time: 15 s
- Cyclic oxidation
  - 1300°C 25min
- XRD
- SEM
- EDS
3. Results & Discussion

- **HfZrTiTa alloy**

  - Equiaxed grains
  
  - BCC single phase structure with near equal atomic percent
3. Results & Discussion

- **HfZrTiTa alloy**

1000°C for 10h, then cooled to room temperature with 10°C/min

![X-ray diffraction pattern](image1.png)

- *as-annealed HfZrTiTa*
  - ▲ BCC
  - ▼ HCP

![Micrograph](image2.png)

- Grain boundary → precipitation enriched with Ta and depleted with Ti, Zr and Hf

**Single BCC → BCC+HCP**
## 3. Results & Discussion

### Pack cementation process

Table 1: The results of pack cementation process

<table>
<thead>
<tr>
<th>Loading amount</th>
<th>Carburizing agent</th>
<th>Carburized layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Full</td>
<td>90% C+10% Na$_2$CO$_3$</td>
<td>×</td>
</tr>
<tr>
<td>Full</td>
<td>90% C+10% CaCO$_3$</td>
<td>×</td>
</tr>
<tr>
<td>Full</td>
<td>100% C</td>
<td>×</td>
</tr>
<tr>
<td>Half full</td>
<td>90% C+10% Na$_2$CO$_3$</td>
<td>×</td>
</tr>
<tr>
<td>Half full</td>
<td>90% C+10% CaCO$_3$</td>
<td>×</td>
</tr>
<tr>
<td>Half full</td>
<td>100% C</td>
<td>√</td>
</tr>
</tbody>
</table>

C+O$_2$=CO$_2$  \[\text{CO}_2+C=2\text{CO}\]
3. Results & Discussion

- Carburized HfZrTiTa

Morphology

HfZrTiTa

100% C → 900°C, 10h

Cutting

Carburized HfZrTiTa

Carburized layer

Cross sectional morphology

Inner layer

Outer layer

07/APR/17
3. Results & Discussion

- **Carburized HfZrTiTa**
  - XRD

  - No obvious peaks
  - Amorphous structure
  - HEA based supersaturated solid solution containing C

XRD pattern of the carburized HfZrTiTa
3. Results & Discussion

- Carburized HfZrTiTa
  - Elemental analysis

- Uniform element distribution in inner carburized layer
- The surface is rich in Ti and C
- The substrate adjacent to the carburized layer exhibits an inhomogeneous composition

EDS line-scanning of the sample
3. Results & Discussion

- Carburized HfZrTiTa

- Elemental analysis

- A: Ta-rich precipitates
- B: Similar element distribution as the initial alloy
- C: Ti-rich region

Element distribution in different regions
3. Results & Discussion

- **Carburized HfZrTiTa**
  - Micro-hardness
  - The maximum hardness was ~1590 HV
  - The average hardness was ~1341 HV
  - The substrate adjacent to the carburized layer was harder than that of HfZrTiTa (~500HV)

The micro-hardness values and corresponding indentations
3. Results & Discussion

- Carburized HfZrTiTa
  - Micro-hardness

- The measured value is lower than the “rule-of-mixtures” average and that of each individual carbide
- The carburized coating could be a HEA based C containing supersaturated solid solution
- The hardness is mainly attributed to the solid solution strengthening

Comparison of Vicker micro-hardness of carburized layer with the reported values
3. Results & Discussion

**Carburized HfZrTiTa**

- Oxidation resistance

![Morphology evolution during oxidation](image)

0.606 mg/(cm²·min)

Weight gain VS oxidation time

Morphology evolution during oxidation
3. Results & Discussion

**Carburized HfZrTiTa**

– Oxidation resistance

The cross-sectional morphology of oxidized sample

Element distribution in different regions after the oxidation test

- Hf
- Zr
- Ti
- Ta
4. Conclusions

- A carburized coating with amorphous structure was produced by solid carburization of HfZrTiTa HEA using 100% graphite powder at 900°C for 10 hrs.

- The carburized coating could be a HEA based C containing supersaturated solid solution according to the micro-hardness, XRD and elemental analysis results.

- The poor oxidation resistance may be caused by the large internal stress generated during the oxidation and the quite low oxidation temperature adopted.
Acknowledgments

- National Natural Science Foundation of China (Grant No. 51501224 & 51371196)
- The colleagues and students in our group
- The organizers of the conference
Thanks for your attention!