Entropy effects on mechanical properties of high entropy alloy CrMnFeCoNi at high and low temperatures

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Overview

“Entropy Effects on Mechanical Properties of Single-Phase High Entropy Alloys”

Fabrication Cantor alloy as single-crystal
Specific heat capacity → Entropy determination
- Tensile & creep testing
- Elastic properties
SEM investigations

Fabrication bcc + hcp as single-crystal
Compression testing
SEM & TEM investigations
Goals of the Project

Central scientific questions to be answered for:

1. Mechanical properties of HEA

Depending on:
- Temperature (low → high)
- Slip system
- Composition

2. Localization dislocation

3. Experimental determination of entropy
Alloy manufacturing

Arc-melting furnace
- Vacuum: 10^{-5} mbar
- Argon atmosphere
- Tungsten-kathode
- Cu-chillplate

Initial weight ~50 g
Cr, Mn, Fe, Co, Ni

Pre-alloyed buttons polycrystalline (CC)

Induction casting of single-crystal (SX) Cantor

Quality check of element content: μ-XRF

University Bayreuth
Christian Gadelmeier, Metals and Alloys
Single-crystal fabrication

Arc melting
Master Alloys

Single crystal casting

Wax model
Ceramic mould

Induction furnace
- Vacuum 10^{-4} mbar
- Argon atmosphere
- Pull down speed 0.5-10 mm/min

Single crystal

Heat treatment of the samples (vacuum)

Determination of $c_p$ via differential scanning calorimetry

Mechanical testing

wire-EDM
Mechanical testing
from -269 °C to ~1300 °C

**Ultrasound resonant spectroscopy**
- Tensor of elastic constants
- Temperature range: RT to ~1300 °C

**Tensile and creep testing** (on air, vacuum or protective gas)
- Tensile test
- Creep testing

TU Dresden ↔ Metals and Alloys, Bayreuth

4x5x6 mm³

27x7x1 mm³
Measuring of specific heat capacity and determination of entropy

Two Netzsch DSC instruments used:
- 
  - 170 °C → +600 °C (Metals and Alloys Bayreuth – DSC 204)
  - room temperature → melting point (Netzsch Selb – DSC 404)
  - Cooling / heating rate 20 K/ min
  - Liquid nitrogen as cooling agent

\[ \text{Enthalpy } T \cdot S = T \cdot (S_{\text{conf}} + S_{\text{th}}) \]

\[ S_{\text{th}} = \int_{0K}^{T} c_p(T') \cdot \frac{1}{T'} dT' \]

\[ \Delta(T \cdot S) = 21 \text{ kJ/mol} \]

Melting interval

\[ \Delta(T \cdot S) = 21 \text{ kJ/mol} \]

Melting enthalpy of Cantor alloy-SX (13.3 kJ/mol)

nickel-SX (15 kJ/mol)
Preliminary Investigations

1. Single-crystal casting under **vacuum** → Mn concentration is nonconstant

   (optimizing)

2. Variation of Mn content under **argon** → low loss of Mn content

   (transfer)

3. Poly-crystal casting under **argon** → constant Mn concentration
1. Single-Crystal under vacuum

Single-crystal

Manufactured: Bridgman furnace
Atmosphere: vacuum
Cast temp.: 1400 °C
Initial weight: 300 g
Pull-down speed: 3 mm/min

nominal: 20 at.% each element

Position D → E: μ-XRF linescan

growth direction of the SX

set value 20 at.% each element

Mn
Ni
Co
Fe
element content in at.%

length linescan μ-XRF in mm

→ Optimizing: variation of Mn, argon
2. Variation of Mn content

Poly-crystal

Manufactured: arc melting
Atmosphere: argon
Initial weight: 50 g

increase of Mn - 20 to 30 at.%

<table>
<thead>
<tr>
<th>target value at.%</th>
<th>20.0</th>
<th>21.0</th>
<th>22.0</th>
<th>25.0</th>
<th>30.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>actual value at.%</td>
<td>19.5 ± 0.5</td>
<td>20.0 ± 0.2</td>
<td>21.0 ± 0.2</td>
<td>24.0 ± 0.3</td>
<td>28.3 ± 0.3</td>
</tr>
<tr>
<td>difference at.%</td>
<td>0.5</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.7</td>
</tr>
</tbody>
</table>

→ Transfer: Poly-crystal production using induction casting / argon atmosphere
3. Poly-Crystal under argon

Poly-crystal
Manufactured: induction casting
Atmosphere: argon
Casting mould: Copper
Initial weight: 250 g

20+2 at.% Mn \rightarrow for balance

μ-XRF linescan - Position A \rightarrow E

max. 21.2 at.% (Mn)
min. 20.4 at.% (Mn)

- - - set value for Mn 20 at.%

no heavy loss of Mn

element content constant

SEM-EDS microsection

<table>
<thead>
<tr>
<th>Mn at.%</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
</tr>
</thead>
<tbody>
<tr>
<td>average</td>
<td>21.1</td>
<td>20.8</td>
<td>20.7</td>
<td>20.8</td>
</tr>
<tr>
<td>deviation</td>
<td>0.3</td>
<td>0.1</td>
<td>0.1</td>
<td>0.2</td>
</tr>
</tbody>
</table>

\rightarrow next step: SX production
Conclusion & Outlook

- First $c_p$ measurements of SX Cantor-alloy realized $\rightarrow$ Entropy $S_{th}$ determined
- Using argon as atmosphere for fabrication $\rightarrow$ stable melting process
- Best element distribution at an initial weight of Mn of 21-22 at.%
- Master alloy fabrication of Cantor alloy has already been started

Outlook

- Current studies: first tensile tests of SX Cantor alloy at low temperature ($\rightarrow$ TU Dresden)
- Fabrication of SX Cantor alloy for HT-characterization will start next
  $\rightarrow$ Mechanical properties: - variation of the composition
  - SX in comparison to PX
Thank you for your attention!