The Effect of Alloy Formulation, Inclusion Content and Cold Work on Void Formation in NiTi Alloys

By

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Outline

- Background
- Materials
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Background: Rahim et al., Acta Materialia 61 (2013) 3667-3686

Fig. 6. SEM back-scatter micrographs of particles/inclusions and pre-existing voids. (a) carbide in C-rich material. (b) oxide in O-rich material. (c) PVA (carbide with crack-like void highlighted by white arrow) in C-rich material. (d) PVA (three oxides with crack-like void highlighted by white arrows) in O-rich material. Black arrows indicate the wire drawing direction.
SEM assessment of crack initiation sites. The double-tip arrows in (e, f) show the drawing direction (e) carbide associated with a crack-like void (view onto wire surface) and (f) oxides associated with crack-like voids (view onto wire surface).
SMST 2014: Inclusions and Voids in $A_s = -25^\circ$C, Cold Drawn Wire

**Carbides**

![Graph showing Carbides]

**Oxides**

![Graph showing Oxides]

**Voids**

![Graph showing Voids]

**Inclusions**

![Graph showing Inclusions]
SMST 2014: Inclusions and Voids in A$_s = +95^\circ$C Cold Drawn Wire

**Carbides**

**Total Cold Reduction, %**
- Maximum
- Average
- Minimum

**Voids**

**Total Cold Reduction, %**
- Maximum
- Average
- Minimum

**Oxides**

**Total Cold Reduction, %**
- Maximum
- Average
- Minimum

**Inclusions**

**Total Cold Reduction, %**
- Maximum
- Average
- Minimum
Procedures

- Two alloys: $A_s = -25^\circ C$ (50.9 atom % Ni); $A_s = +95^\circ C$ (49.8 atom % Ni).
- Samples: hot rolled 6.35 mm, wires cold drawn in steps from 5.99 mm to 0.53 mm
- 10% to 99.3% cold reduction with intermediate anneals.
- 3 samples for each wire size, 9 fields per sample.
- Measured all inclusions greater than 0.12 $\mu m$.
- Optical microscopy in longitudinal centerline plane in two steps: focused on inclusions, focused on voids.
- Counted carbides, oxides and voids separately, however:
  - Oxide containing enveloped carbide is counted as oxide only.
  - Maximum dimension of carbide or oxide per F2063 – 12 is the “maximum length of all contiguous particles and voids, including particles separated [only] by voids.” Therefore, the maximum dimension measures contiguous and included voids.
- Analyzed data versus total cumulative cold work.
- Analyzed cumulative total area of all inclusions and voids for all fields.
- Analyzed cumulative total length of all inclusions and voids for all fields.
- Reporting trends in maximum inclusion size and area.
$A_s = -25^\circ C$ Alloy 6.35 mm Hot Rolled Coil

Focus on inclusions

Focus on voids
$A_s = -25^\circ C$ Alloy 2.16 mm Wire

Focus on inclusions

Focus on voids
$A_s = -25^\circ C$ Alloy 0.53 mm Wire

Focus on inclusions

Focus on voids
Cumulative Area of Inclusions and Voids in $A_s = -25^\circ C$ Alloy
Cumulative Length of Inclusions and Voids in $A_s = -25^\circ$C Alloy
Maximum Area Fraction of Inclusions and Voids in $A_s = -25^\circ C$ Alloy
Observations on $A_s = -25^\circ C$ Alloy

- Total area fraction of inclusions increases slightly through multiple steps of cold drawing.
- Total inclusion length increases slightly in cold drawing.
- The increase in area and length is associated with void formation.
- Void formation occurs at both carbides and oxides.
- Maximum inclusion size as defined by ASTM F2063 is associated with the formation of stringers of contiguous inclusions and voids.
- The maximum inclusion area and length remain below 1.0% and 17 µm in $A_s = -25^\circ C$ alloy wire at 0.53 mm diameter.
$A_s = +95^\circ$C Alloy Hot Rolled Coil

Focus on inclusions

Focus on voids
$A_s = +95^\circ C$ Alloy 2.16 mm Wire

Focus on inclusions

Focus on voids
\[ A_s = +95^\circ C \text{ Alloy 0.53 mm Wire} \]

Focus on inclusions

Focus on voids
Cumulative Area of Inclusions and Voids in $A_s = +95^\circ C$ Alloy
Cumulative Length of Inclusions and Voids in $A_S = +95^\circ C$ Alloy
Maximum Area Fraction of Inclusions and Voids in $A_s = +95^\circ C$ Alloy
Observations for $A_s = +95^\circ\text{C}$ Alloy

- Total inclusion area initially increases and then decreases through multiple steps of cold drawing. The increase is associated with increased void content due to the fracture of inclusions.
- Total inclusion length increases through cold drawing.
- Void formation is associated primarily with oxides.
- Void formation occurs frequently at an oxide – carbide interface.
- Maximum inclusion length is the result of the formation of stringers of contiguous fractured inclusions and voids.
- The scale of void formation is larger in the low Ni-Ti ratio alloy which contains more intermetallic oxide.
- The maximum inclusion area and length remain below 2.0% and 150 µm in $A_s = +95^\circ\text{C}$ alloy wire at 0.53 mm diameter.
Conclusions

- The trends in inclusion area and length are similar to the trends in void formation and reduction.
- In cold drawing, inclusion size and area initially increase as inclusions are fractured and aligned in the drawing direction with the concurrent formation of included and contiguous voids.
- With continued cold drawing, void content is reduced resulting in a reduction in the size and area of inclusions.
- The reduction of the radius of the product increases the fraction of the original diameter of the product evaluated in a fixed field of view. The initial increase in area fraction of inclusions is due, in part, to the radial compression of the interdendritic pattern of inclusions in the wire.
- For NiTi alloys, observations made on higher inclusion content alloy (49.8 a/o Ni) can lead to a better understanding of the behavior of lower inclusion content alloy (50.9 a/o Ni).
- This study was not able to identify the mechanism for void reduction.
- An experimental program with smaller cold reduction steps may be able to elucidate the mechanism for void reduction.
Thanks for your attention