## Structural Material

*"OAK

- RIDGE


# Powder Metallurgy - Hot Isostatic Pressing of Steels in Support of Microreactors 

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## Background

- Powder metallurgy hot isostatic pressing (PM-HIP) is a manufacturing method that produces components by consolidating metal powder
- Minimizes additional fabrication steps
- Eliminates solidification structures
- Eliminates directional grain elongation caused by rolling or forging


MTC Powder Solutions


UK - Nuclear Advanced Manufacturing Research Center (UK-NAMRC) System

## PM-HIP Adoption for Microreactors

- PM-HIP may benefit structural components for microreactors (i.e., core barrels, primary coolant loops, etc.) by reducing construction time, reducing waste, and improving component availability


## Goals

- Demonstrate high temperature mechanical properties of PM-HIP compared to wrought materials for Sec. III Div. 5 structural alloys
- Address PM-HIP 316 stainless steels to support multiple advanced reactors
- Develop specifications and acceptance criteria for PM-HIP components
- Low temperature code case (up to $371^{\circ} \mathrm{C}$ )
- High temperature code case ( $371^{\circ} \mathrm{C}<\mathrm{T}<816^{\circ} \mathrm{C}$ )



## PM-HIP Div. 5 Code Case Roadmap

| Scoping mechanical testing of PM-HIP alloys for Sec. III, Div. 5 qualification | Review results of scoping study | Scoping results are promising <br> Scoping study results are not promising | Identify PM-HIP alloys to be qualified in Sec. III, Div. 5 | Generate data for ASTM PM-HIP material specification development and ASME BPVC Section III, Div. 5 qualification |
| :---: | :---: | :---: | :---: | :---: |
| Sources of Data INL, UKNAMRC <br> Potential alloys: <br> 316H, Alloy 800H, Alloy 617, Gr 91, Alloy 709 | Task Group on Div. 5 AM <br> Components |  | Task Group on Div. 5 AM <br> Components | Responsible <br> Party TBD |

## PM-HIP Div. 5 Code Case Roadmap



## Materials - 316 SS

| Powder Compositions (wt\%) |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | C | Ni | Cr | Mo | Ti | AI | Si | Mn | S | P | N | 0 |
| $\begin{gathered} \text { 316H } \\ \text { Billet } \mathbf{1}^{11} \end{gathered}$ | 0.055 | 11.8 | 16.3 | 2.51 | 0.01 | 0.01 | 0.18 | 0.22 | 0.01 | 0.003 | 0.140 | 0.0167* |
| $\begin{gathered} 316 \mathrm{H} \\ \text { UK-NAMRC² } \end{gathered}$ | 0.05 | 11.9 | 17.1 | 2.52 | <0.01 | 0.01 | 0.17 | 0.18 | 0.002 | 0.004 | 0.076 | 0.0093* |
| $\begin{gathered} 316 \mathrm{~L} \\ \text { UK-NAMRC² } \end{gathered}$ | 0.015 | 11.9 | 17.7 | 2.44 | 0.003 | 0.006 | 0.83 | 1.88 | 0.008 | 0.008 | 0.060 | 0.0117** |


| Consolidated Product Chemical Compositions (wt\%) |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | C | Ni | Cr | Mo | Ti | AI | Si | Mn | S | P | N | 0 |
| $\begin{gathered} \text { 316H } \\ \text { Billet } 1^{11} \end{gathered}$ | 0.040 | 12.0 | 16.4 | 2.48 | 0.005 | 0.007 | 0.17 | 0.21 | 0.003 | 0.002 | 0.147 | 0.020 |
| $\begin{gathered} 316 \mathrm{H} \\ \text { UK-NAMRC² } \end{gathered}$ | 0.040 | 11.8 | 17.3 | 2.53 | <0.01 | <0.01 | 0.17 | 0.18 | <0.003 | <0.005 | 0.069 | 0.015 |
| $\begin{gathered} \text { 316L } \\ \text { Billet } 1^{3} \end{gathered}$ | 0.012 | 11.5 | 17.4 | 2.22 | 0.006 | <0.002 | 0.65 | 0.58 | 0.009 | 0.011 | 0.049 | 0.020 |
| $\begin{gathered} \text { SA } 240 \\ \text { S31609 (316H) } \end{gathered}$ | $\begin{gathered} 0.04- \\ 0.10 \end{gathered}$ | $\begin{aligned} & 10.0- \\ & 14.0 \end{aligned}$ | $\begin{aligned} & 16.0- \\ & 18.0 \end{aligned}$ | $\begin{aligned} & 2.00- \\ & 3.00 \end{aligned}$ | - | - | 1.00 | 2.00 | 0.030 | 0.045 |  | - |
| ASME III Div. $5\left(>595^{\circ} \mathrm{C}\right)$ | $\geq 0.04$ | $\begin{aligned} & 10.0- \\ & 14.0 \end{aligned}$ | $\begin{aligned} & 16.0- \\ & 18.0 \end{aligned}$ | $\begin{aligned} & 2.00- \\ & 3.00 \end{aligned}$ | 0.04 | 0.03 | 1.00 | 2.00 | 0.030 | 0.045 | $\geq 0.05$ | - |

## Results - 316 SS Microstructure

316H - Billet 1


$$
\begin{gathered}
\mathrm{d}_{\mathrm{avg}}=35 \mu \mathrm{~m} \\
\mathrm{HV}_{0.3}=224
\end{gathered}
$$

316H - UK-NAMRC


$$
\begin{gathered}
\mathrm{d}_{\mathrm{avg}}=47 \mu \mathrm{~m} \\
\mathrm{HV}_{0.3}=194
\end{gathered}
$$

*SA240: ASTM No. 7 ( $\mathrm{d}=31.8 \mu \mathrm{~m}$ ) or coarser *Sec. II Part A: $\leq 200$ HV

## Results - 316 SS Oxide Analysis

316H - Billet 1


Oxide Area Fraction $=0.15 \%$

316H - UK-NAMRC


Oxide Area Fraction $=0.37 \%$


## Results - 316 SS Oxide Analysis

- Qualitative EDS Analysis - UKNAMRC 316H


| 5 rm | Ni |  |
| :---: | :---: | :---: |
|  |  |  |
|  |  |  |
|  |  |  |



## Procedures - Fatigue Testing

$$
650^{\circ} \mathrm{C}, \Delta \varepsilon=1 \%, \quad R=-1, \quad \dot{\varepsilon}=0.001 \mathrm{~s}^{-1}
$$

Low cycle fatigue (LCF)


Creep-fatigue (CF) 30 min . Hold



$$
r=\left|\frac{\sigma_{\text {tensile }}}{\sigma_{\text {compression }}}\right|
$$

## Results - 316L Billet 1

- $650^{\circ} \mathrm{C}, \Delta \varepsilon=1 \%, R=-1, \dot{\varepsilon}=0.001 \mathrm{~s}^{-1}$






## Results - 316L UK-NAMRC

- $650^{\circ} \mathrm{C}, \Delta \varepsilon=1 \%, R=-1, \dot{\varepsilon}=0.001 \mathrm{~s}^{-1}$






## Results - 316H Billet 1

- $650^{\circ} \mathrm{C}, \Delta \varepsilon=1 \%, R=-1, \dot{\varepsilon}=0.001 s^{-1}$





MRP

## Results - 316H UK-NAMRC

- $650^{\circ} \mathrm{C}, \Delta \varepsilon=1 \%, R=-1, \dot{\varepsilon}=0.001 s^{-1}$






## Results - Wrought vs. PM-HIP



Oxygen Comparison for PM-HIP Materials

*Note: excludes other compositional and grain size influence

## Results - Crack Morphologies




## Results - Crack Morphologies

- Creep-Fatigue 316H UK-NAMRC



## Results - Crack Morphologies

- Creep-Fatigue 316H UK-NAMRC



## Conclusions

- PM-HIP 316 stainless steels continued to show reduced cycles to failure under creep-fatigue testing conditions compared to wrought 316 stainless steel
- Grain boundary oxides are likely resulting in reduced creep-fatigue resistance through crack nucleation and propagation
- Microstructure showed grain boundary cavitation ahead of the main crack
- Low cycle fatigue specimens showed transgranular and intergranular crack propagation
- Creep-fatigue specimens only showed intergranular crack nucleation/propagation


## Future Work

- Conduct elevated temperature mechanical testing on 316H with low oxygen and processed using different hot isostatic pressing conditions
- One-third of the powder was hot isostatically pressed and underwent a heat treatment identical to MTC Billet 1
- Another third is being heat treated at different conditions to try to influence the oxide size/distribution

| Powder Composition $(\mathbf{w t \%})$ |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | $\mathbf{N i}$ | $\mathbf{C r}$ | $\mathbf{M o}$ | $\mathbf{C}$ | $\mathbf{S i}$ | $\mathbf{M n}$ | $\mathbf{S}$ | $\mathbf{P}$ | $\mathbf{N}$ | $\mathbf{O}$ |  |  |  |
| 316H Billet 2 | 12.0 | 17.0 | 2.53 | 0.05 | 0.20 | 0.21 | 0.003 | 0.004 | 0.101 | $0.0120^{*}$ |  |  |  |



