

Impact toughness of commercial and model FeCrAl alloys[☆]Zhiqian Sun^{*}, Yukinori Yamamoto, Xiang Chen

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ABSTRACT

FeCrAl alloys are under development as possible candidate materials of accident-tolerant fuel cladding in light water reactors. In this study, the cracking resistance of FeCrAl alloys was evaluated using half-size Charpy impact tests at temperatures ranging from room temperature to 600 °C. Ingot-metallurgy wrought FeCrAl alloys with base alloy compositions of Fe-(10–13)Cr-6Al-2Mo, in weight percent, and commercial powder-metallurgy FeCrAl alloys (Kanthal APMT) were investigated. All studied alloys showed almost zero absorbed impact energy at room temperature with brittle fracture behavior. The as-received APMT alloys exhibited poor impact toughness even at 600 °C. The ductile-brittle transition temperatures of the wrought FeCrAl alloys varied from 119 to 318 °C. The possible effects of microstructures, residual strain, materials preparation methods, process conditions, and chemical composition on the impact toughness of FeCrAl alloys were investigated by examining the Charpy impact data in combination with microstructural details of the alloys.

1. Introduction

Iron-chromium-aluminum (FeCrAl) alloys are extremely resistant to oxidation at elevated temperatures because of the formation of a protective alumina layer on the outer surface [1–3], making them highly suitable for industrial and other heating applications. Recently, FeCrAl alloys are under development to replace current commercial zirconium alloys as accident-tolerant fuel cladding in light water reactors (LWRs) in an effort to gain greater margins of safety in the event of an accident [4–9].

The fuel cladding in LWRs is usually ~4 m in length and has an outside diameter of ~10 mm [10]. FeCrAl alloys show larger neutron absorption than zirconium alloys, which limits the wall thickness of the FeCrAl cladding to less than ~0.5 mm [11]. A combination of several processing steps is required to produce final FeCrAl tubes with the desired dimensions and microstructure [12]. Because of the limited formability of FeCrAl alloys [13–15], their resistance to crack initiation and propagation is critical for successful tube fabrication. In previous fabrication trials [12,16,17], cracks along the longitudinal axis formed easily and propagated through FeCrAl tube wall during tube drawing especially when residual strain existed. Inter-pass annealing has been applied to reduce residual strain and restore the processability of

FeCrAl alloys [15]. The resistance to cracking also plays an important role in transporting the cladding as well as the in-service performance of FeCrAl alloys, since it is critical that the hermeticity of the cladding be maintained to avoid the release of radionuclides. However, few studies have investigated the cracking resistance of FeCrAl alloys [18,19].

This study evaluates the cracking resistance of FeCrAl alloys using Charpy impact tests at temperatures ranging from room temperature (RT) to 600 °C. Commercial powder-metallurgy (PM) FeCrAl alloys, i.e., Kanthal APMT, and wrought ingot-metallurgy (IM) FeCrAl alloys were selected for testing. The effects of microstructures, residual strain, alloy preparation methods, process conditions, and chemical composition on the impact toughness of these alloys are discussed. The results of this study provide future directions for optimizing alloy and process development to achieve improved cracking resistance in FeCrAl alloys.

2. Materials and experimental

2.1. Material preparation

Table 1 lists the compositions of the studied alloys. The compositions of the wrought FeCrAl alloys were based on Fe-(10–13)Cr-6Al-

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Table 1
Analyzed compositions of the studied alloys.

ID	Composition ^a (wt%)											
	Fe	Cr	Al	Y	Mo	Si	Hf	Mn	Ni	Ta	Zr	
APMT-heat #1	Bal.	21.26	4.85	0.21	3.03	0.29	0.23	0.27	0.27	0.13	0.12	
APMT-heat #2	Bal.	21.27	4.80	0.22	3.00	0.14	0.29	0.30	0.23	0.01	0.12	
IM-APMT	Bal.	21.26	4.52	–	2.81	0.42	0.25	0.17	0.27	–	0.10	
10Cr	Bal.	9.88	6.03	0.05	1.97	0.21	–	–	–	–	–	
12Cr	Bal.	12.18	6.11	0.04	2.04	0.2	–	–	–	–	–	
13Cr	Bal.	13.00	6.29	0.06	1.99	0.2	–	–	–	–	–	

ID	Composition ^a (wt%)			
	C	S	O	N
APMT-heat #1	0.035	0.0005	0.0572	0.0424
APMT-heat #2	0.039	0.0003	0.0659	0.0424
IM-APMT	0.039	0.0020	0.0019	0.0416
10Cr	0.003	0.0005	0.0034	0.0004
12Cr	< 0.01	< 0.005	–	–
13Cr	0.001	< 0.0003	0.0010	0.0004

^a Measured by induction coupled plasma optical emission spectroscopy (most of the elements), combustion analysis (C and S), and inert gas fusion analysis (O and N).

2Mo, weight percent (wt%), and the Cr content was used to identify different wrought FeCrAl alloys (i.e., 10Cr, 12Cr, and 13Cr). Heats of 10Cr, 12Cr, and 13Cr were prepared using a commercial vacuum induction melt process. Two heats of the 13Cr alloy with almost identical compositions were prepared, and Table 1 only shows one of them. As indicated in Table 1, the compositions of the commercial APMT alloys were more complex and contained many minor alloying elements or impurities (e.g., Y, O, C, N, Hf, Mn, and Ni) compared with the wrought FeCrAl alloys. The wrought FeCrAl alloys simply consisted of a solid-solution body-center-cubic (bcc) matrix and a few Y-enriched particles, while the APMT alloys had many more secondary particles (e.g., oxides, carbides, and nitrides [20]). Detailed characterization of the particles in APMT is beyond the scope of this study. Various thermomechanical treatments were employed to control the microstructures of the studied alloys. The detailed process history of each specimen is listed below.

- APMT-heats #1 and #2: Both heats were as-received materials in the form of either a square bar (#1) or a rod with ~12 mm in diameter (#2), produced through unknown production routes (probably formed by extrusion or forging).
- APMT-heat #1-HR800: As shown in Section 3.1.1, APMT-heat #1 exhibited an inhomogeneous bimodal grain structure with considerable residual strain. To control the microstructure, APMT-heat #1 was hot-forged at 800 °C with a ~25% thickness reduction (50.8 → 38.1 mm), hot-rolled at 800 °C with a total ~83% thickness reduction (38.1 → 6.4 mm), and then annealed at 800 °C for 30 min. Further annealing at 1000 °C for 30 min was applied to obtain a fully recrystallized grain structure.
- IM-APMT: To investigate the possible effects of material production routes on the impact toughness of FeCrAl alloys, IM-APMT was prepared with a target composition similar to those of the as-received APMT heats #1 and #2 using arc-melting and thermo-mechanical treatments. Lower amounts of Y and O in IM-APMT compared with the as-received APMT heats were probably because of formation of yttrium oxides as slags during arc-melting process. After homogenization at 1200 °C for 2 h, the ingot was hot-forged at 800 °C with a ~43% thickness reduction (44.5 → 25.4 mm) and then hot-rolled at 800 °C with an additional ~75% thickness reduction (25.4 → 6.4 mm), followed by annealing at 800 °C for 1 h.
- 10Cr-HR800 and 13Cr-HR800: After homogenization at 1200 °C for 4 h, the ingots were hot-rolled at 800 °C followed by annealing at 800 °C for 4 h. The total thickness reductions of 10Cr-HR and 13Cr-

HR were ~67% (19.1 → 6.4 mm) and 50% (12.7 → 6.4 mm), respectively.

- 10Cr-HR650, 12Cr-HR650, and 13Cr-HR650: After homogenization at 1200 °C for 4 h (10Cr and 13Cr) or being hot-isostatically pressed at 1200 °C and 100 MPa for 4 h (12Cr), the ingots were hot-forged at 1050 °C (10Cr and 13Cr) or 800 °C (12Cr) with a total ~79% thickness reduction (88.9 → 19.1 mm). The forged plates were annealed for 30 min at 800 °C and then hot-rolled at 650 °C with inter-pass annealing at 800 °C for 15 min with a target thickness of 7.6 mm (~60% thickness reduction). The rolled plates were annealed at 800 °C for 30 min. The plates of 12Cr and 13Cr were then warm-rolled at 300 °C with a ~10% thickness reduction to study the effects of residual strain on the impact toughness of FeCrAl alloys.
- 13Cr-HR650-ANN: Additional annealing at 800 °C for 15 min was applied to 13Cr-HR650 to eliminate the effects of deformation and restore the material to an annealed condition.

It should be noted that all hot-rolling or warm-rolling were performed with ~10% thickness reduction per pass, and inter-pass annealing for 10–15 min at corresponding temperatures (unless otherwise specified) was applied during hot-rolling.

2.2. Microstructure characterization

Metallographic samples were sectioned by electrical discharge machining (EDM) and then mounted into an epoxy resin. After grinding, these samples were polished using a Buehler's VibroMet polisher with 0.3 μm Al₂O₃ and subsequent 0.1 μm colloidal silica. Electron back-scattered diffraction (EBSD) data were collected using a JEOL JSM-6500F scanning electron microscope and were analyzed using EDAX's OIM data analysis software package. All displayed micrographs were centered relative to their thickness or radial direction, unless otherwise specified. For rolled plates, micrographs were taken from the transverse direction (TD) with the rolling direction (RD) parallel to the vertical axis. Multiple scans were performed along the normal direction (ND) to identify the microstructural evolution through the rolled-plate thickness. The scanning step sizes were in the range from 0.8 to 3 μm. The EBSD data was cleaned up using the "grain dilation" procedure with a grain tolerance angle of 5° and a minimum grain size of ~5 μm. Colors in orientation maps of rolled plates represented corresponding grain orientations relative to the plate ND, based on the color triangle. High-angle boundaries with misorientations larger than 15° were superimposed on orientation maps as black lines. Recrystallized grains were identified as those having average grain sizes larger than ~3 μm with a grain tolerance angle of 0.5°. The calculation of average grain sizes was weighted by grain areas based on the EBSD results. The kernel average misorientation (KAM) was employed to qualitatively characterize residual strain in FeCrAl alloys. A kernel is defined as a group of scanning points surrounding a center point with a prescribed size (n^{th} nearest neighbor). The KAM of a kernel, whose value was assigned to its center point, was calculated by averaging the misorientations (smaller than a tolerance value) between the center point and all perimeter points. A first-nearest-neighbor kernel was used with a misorientation tolerance value of 5°. The texture information, including inverse pole figures (IPFs) and orientation distribution function (ODF) maps, was constructed using the spherical harmonics method [21] with a series rank of 16 and a Gaussian half width of 5°. An orientation tolerance of 15° was used for estimating the area fractions of certain orientations and texture fibers. The grain orientation $\{hkl\} <uvw>$ in a rolled plate was specified as $\{hkl\}$ parallel to the rolling plane and $<uvw>$ parallel to the RD.

2.3. Mechanical tests

The Vickers hardness tests were performed using a Shimadzu HMV-G hardness tester with 0.5 kg weight force and 10 s dwell time. At least

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