



# Processability evaluation of a Mo-containing FeCrAl alloy for seamless thin-wall tube fabrication<sup>☆</sup>



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## ABSTRACT

The processability of a Mo-containing FeCrAl alloy (Fe-13Cr-5.2Al-2Mo base, in wt%), developed for accident-tolerant nuclear fuel claddings, was evaluated through a stepwise rolling process at 400 °C under two different inter-pass annealing conditions (i.e., 650 °C for 1 h and at 870 °C for 30 min). The inter-pass annealing at 870 °C easily softened the FeCrAl alloy; however, it led to the formation of coarse grains of ~200 μm. On the other hand, the FeCrAl alloy maintained elongated, deformed grains with the inter-pass annealing at 650 °C, but the annealed samples showed relatively high deformation resistance and strong texture. Important aspects concerning the processability and microstructural control of FeCrAl alloys, such as deformation inhomogeneity, texture development, and grain coarsening, were discussed. Optimized processing conditions were recommended, based on the results, to achieve desirable microstructures with balanced processability and mechanical properties.

## 1. Introduction

Iron-chromium-aluminum (FeCrAl) alloys are historically known as an inexpensive Fe-base heating element and used for a long time. Currently, nuclear-grade wrought FeCrAl alloys are under development as one of accident-tolerant fuel (ATF) cladding materials in light-water reactors (LWRs) because of their superior oxidation and corrosion resistance in high-temperature steam environments compared with current commercial zirconium-based alloys [1–6]. FeCrAl alloys usually consist of Fe, Cr, and Al together with other minor elements. The amounts of Cr and Al have been fine-tuned to obtain balanced properties (e.g., mechanical properties and oxidation/corrosion resistance) and sufficient deformability. The amounts of Cr (10–13 wt%) and Al (5–6 wt%) additions in FeCrAl alloys were found to provide improved surface protection effects at both service temperature and elevated temperatures in accident case scenarios. Minor alloying elements (e.g., molybdenum [Mo] and/or niobium [Nb]) were added, focused on improving the strength and controlling the microstructures of FeCrAl alloys [6].

Owing to their high Cr and Al contents, FeCrAl alloys maintain the stable body-centered-cubic (bcc) structure up to their melting points, eliminating the chance of grain refinement through phase

transformation as in carbon steels [7,8]. In addition, bcc-iron materials with Cr and Al additions usually suffer from poor ductility [9,10]. Another consideration is that the fuel cladding in LWRs requires thin-wall seamless tubes ~4 m in length with an outer diameter of ~10 mm and a wall thickness of less than 0.5 mm [11]. These facts impose considerable challenges for fabricating tubes from FeCrAl alloys.

There are two preferred routines for fabricating thin-wall seamless tubes: tube drawing and pilgering. We have successfully fabricated seamless thin-wall FeCrAl tubes with 9.5 mm in outer diameter and 0.38 mm in wall thickness by tube drawing [12], as shown in Fig. 1a. For both methods, a number of passes or reductions are required to achieve the final dimensions. It is important to apply inter-pass annealing after each reduction step for FeCrAl alloys to restore their deformability, because of a strong work-hardening of FeCrAl alloys [13] prohibiting continuous reduction processes without premature failures. Proper processing conditions (i.e., reduction per pass and inter-pass annealing temperature and time) lead to desirable microstructures not only for processability but also for the mechanical properties of final tube products. Fig. 1b shows a cross-section optical micrograph of a FeCrAl tube [12], exhibiting coarse grains with a size of ~100 μm. Such coarse grain structure is not ideal for the tube fabrication process because it could lead to nonuniform reductions of wall thickness due to

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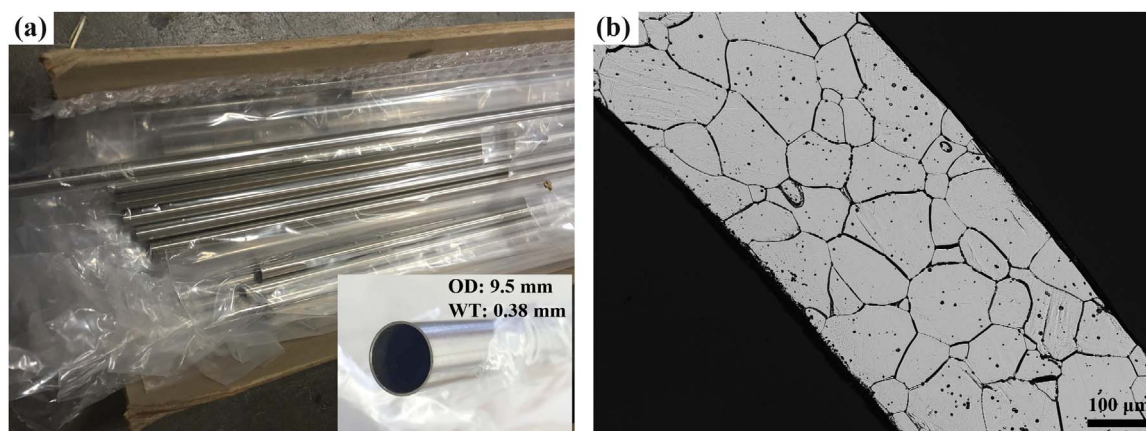


Fig. 1. (a) FeCrAl tube fabricated by tube drawing; (b) cross-section optical micrograph of a FeCrAl tube.

inhomogeneous deformation of individual grains. Besides, those coarse grains are likely to compromise the mechanical properties (e.g., tensile strength and fatigue resistance) of final tubes. Field et al. [14] also indicated that the grain structure is also important for determining the radiation tolerance of FeCrAl alloys. For these reasons, proper control of grain structure by selecting optimized processing conditions is critical for successful fabrication of thin-wall FeCrAl tubes.

In this study, the processability of a Mo-containing FeCrAl alloy, C35M4 (Fe-13Cr-5.2Al-2Mo base, in wt%), was evaluated through a stepwise warm rolling process at 400 °C with two different inter-pass annealing conditions (i.e., 650 °C for 1 h and 870 °C for 30 min). The microstructure and mechanical properties evolution of C35M4 during stepwise rolling were investigated. Moreover, issues such as deformation heterogeneity on the grain scale, texture development, and grain coarsening were discussed. The results further our understanding of deformation in FeCrAl alloys and provide guidelines to optimize the processability of FeCrAl alloys while maintaining desirable mechanical properties.

## 2. Materials and methods

A columnar-shape ingot ~100 mm in diameter × 430 mm in length was prepared by vacuum induction melting. Table 1 lists its analyzed composition. Notice that carbon was not intentionally added because of very little carbon dissolution in the bcc matrix which would result in no significant effect on the strengthening and microstructure control expected through the controlled carbide precipitation [6]. The ingot was homogenized at 1200 °C in argon cover gas for 4 h, extruded at 1050 °C with an area reduction ratio of 9.6:1, and then annealed at 800 °C for 30 min. The annealed bar showed nearly equiaxed recrystallized grains with an average size of ~70 μm. Two plates with an initial thickness of 3.7 mm were cut from the bar, with the longitudinal axis parallel to the extrusion direction, by electric discharge machining (EDM).

Both plates were stepwise warm-rolled at 400 °C with inter-pass annealing simulating tube drawing process, to investigate microstructure evolution and mechanical-properties during process. Two annealing conditions were used: (A) 650 °C for 1 h and (B) 870 °C for 30 min. A total 16 passes were applied with a nominal 10% thickness reduction per pass. EDM was used to cut a coupon from the rolled plates after each pass to track the microstructure and mechanical properties. The measured thickness reductions per pass were in the range of

Table 1  
Analyzed composition of C35M4 in wt%.

Fe	Cr	Al	Y	Mo	Si	C	S
79.56	12.89	5.25	0.04	2.06	0.20	< 0.01	0.001

6.5–10.3%, achieving ~77% total thickness reduction (the final thickness was ~0.85 mm) after 16 passes. In the following text, as-rolled (annealed) samples after the  $n^{\text{th}}$  pass will be referred to as “An” (An’) or “Bn” (Bn’), depending on the annealing conditions.

Metallographic samples were sectioned from rolled and annealed plates by EDM and then mounted in epoxy. After grinding, these samples were polished using a Buehler’s VibroMet™ polisher with 0.3 μm Al<sub>2</sub>O<sub>3</sub> and subsequent 0.1 μm colloidal silica. All micrographs were taken from the transverse direction (TD) with the rolling direction (RD) parallel to the vertical axis. Electron backscatter diffraction (EBSD) data were collected using a JEOL 6500 FEG-scanning electron microscope (SEM) and analyzed using EDAX’s OIM™ data analysis software package. Information including the average grain size, grain aspect ratio, kernel average misorientation, and texture was extracted. In inverse pole figure (IPF) maps, the colors of grains indicate the crystal directions along the normal direction (ND). Grain boundaries with misorientation larger than 15° were superimposed in the IPF maps as black lines. Edge grains were included to calculate the average grain size with a grain tolerance angle of 5°. A kernel is defined as a set of scanning points surrounding a center point with a prescribed size ( $n^{\text{th}}$  nearest neighbor). The kernel average misorientation was calculated by averaging the misorientation (smaller than the tolerance value) between the center point of a kernel and all perimeter points in the same kernel. The kernel average misorientation value was assigned to the center point of the kernel. The first-nearest-neighbor kernels were used with a tolerance angle of 5°, unless otherwise specified. A {hkl} <uvw>-orientated grain was designated as having the {hkl} plane parallel to the rolling plane and the <uvw> direction parallel to the RD. The texture pole figures were constructed using the spherical harmonics method [15] with a series rank of 16 and a Gaussian half width of 5°. Backscattered electron imaging in a Hitachi S4800 FE-SEM was used to characterize subgrains.

Vickers hardness and tensile tests were performed on selected samples. The Vickers hardness tests were conducted using a Shimadzu HMV-G hardness tester with 0.5 kg weight force and 10 s dwell time. At least eight measurements were made for each test. Tensile tests were conducted at a strain rate of 10<sup>-3</sup> s<sup>-1</sup> at room temperature. Two kinds of dog-bone shape sheet tensile specimens were used. Specimens (designation SS-3) ~7.6 mm long × 1.4 mm wide × 0.8 mm thick at the gauge section were prepared from the annealed extruded bar. Smaller specimens (designation SS-J3) ~5.0 mm long × 1.2 mm wide × 0.7 mm thick at the gauge section were prepared from the final rolled plates. The tensile axis was parallel to the extrusion direction or the RD.

## 3. Results

C35M4 consisted of a solid-solution bcc matrix with a few yttrium-enriched particles. The Vickers hardness value of C35M4 before

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