

C/ORNL 94-0314

**CRADA Final Report
for
CRADA Number C/ORNL 94-0314**

**Development of Commercial Applications
of a FAPY Alloy**

**V. K. Sikka
Oak Ridge National Laboratory**

**F. Hall
Hoskins Manufacturing Company
Hamburg, Michigan**

Date Published: August 2001

**Prepared by the
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831
Managed by
Lockheed Martin Energy Research Corporation
for the
U.S. Department of Energy
Under contract DE-ACO5-96OR22464**

APPROVED FOR PUBLIC RELEASE

UNLIMITED DISTRIBUTION

CONTENTS

ABSTRACT	1
CRADA OBJECTIVE	1
COMPLETION OF OBJECTIVE	2
CRADA BENEFITS TO DOE.	3
TECHNICAL DISCUSSION	3
REPORT OF INVENTIONS	30
COMMERCIALIZATION POSSIBILITIES	31
PLANS FOR FUTURE COLLABORATION.	31
CONCLUSIONS	31
DISTRIBUTION	33

Abstract

The Fe-16 at. (8.5 wt) % Al alloy, known as FAPY, has been identified as a superior material for heating element applications. However, while the 15-lb heats melted at the Oak Ridge National Laboratory (ORNL) could be processed into wire, the large heat melted at Hoskins Manufacturing Company (Hoskins) could not be processed under commercial processing conditions. The primary objective of the Cooperative Research and Development Agreement (CRADA) was to demonstrate that wire of the FAPY alloy could be produced under commercial conditions from air-induction-melted (AIM) heats. The specific aspects of this CRADA included:

1. Melting 15-lb heats by AIM or vacuum-induction melting (VIM) at ORNL.
2. Development of detailed processing steps including warm drawing and annealing temperature and time during cold-drawing steps.
3. Melting of 1400-lb heats at Hoskins by the Exo-Melt™ process and their chemical analysis and microstructural characterization.
4. Development of tensile properties of sections of ingots from the large heats in the as-cast, hot-worked, and hot- and cold-worked conditions.
5. Microstructural characterization of cast and wrought structures and the fractured specimens.
6. Successful demonstration of processing of AIM heats at Hoskins to heating element wire.

The aspects of this CRADA listed above have demonstrated that the FAPY alloy of the desired composition can be commercially produced by AIM by the use of the Exo-Melt™ process. Furthermore, it also demonstrated that the wire processing steps developed for 15-lb heats at ORNL can be successfully applied to the production of wire from the large AIM heats.

CRADA Objective

The objective of this CRADA was to address the technical issues dealing with commercial-scale melting and processing of a FAPY alloy developed by ORNL. Hoskins has interest in the production of wire of FAPY alloy for heating elements and a range of other applications. Prior to this CRADA, Hoskins had melted large heats of FAPY and hot-rolled its ingots to 0.25-in.-diam rod coils. However, there were questions regarding the ease of cold working of the 0.25-in.-diam rod coils into wires of different sizes by Hoskins' conventional working process.

Research sponsored by the U.S. Department of Energy, Office of Fossil Energy, Advanced Research and Technology Development Materials Program, [DOE/FE AA 15 10 10 0, Work Breakdown Structure Element ORNL-2(H)], under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

The CRADA effort was divided into the following four phases:

Phase 1: Rod Samples

In this phase, Hoskins was to supply 0.25-in.-diam by 20-ft-long rod from their coil for processing at ORNL.

Phase 2: Characterize the Rod

This characterization of the Hoskins rod consisted of three tasks:

Task 2.1: Chemical analysis.

Task 2.2: Mechanical properties testing in the as-received condition and after 1-h treatments at 800, 900, 1000, and 1200°C. All tensile tests were to be done at room temperature.

Task 2.3: Metallography to check the effect of heat treatment on grain size of 0.25-in.-diam rod.

Task 2.4: Letter report to Hoskins of the findings of Tasks 2.1 through 2.3.

Phase 3: Simulation

In this phase, the process at Hoskins was to be simulated at ORNL. The tasks under this phase included:

Task. 3.1: Melting of two 15-lb heats of FAPY alloy by vacuum-induction melting and casting into 3-in.-diam ingots. One ingot was to be used to characterize the properties in the as-cast condition.

Task 3.2: Hot-Extrusion - The second ingot was to be hot extruded at 1050°C from 3- to 0.5-in.-diam rod.

Task 3.3: Mechanical Properties – Specimens from the hot-extruded rods will be room-temperature tensile tested after treatments at 800, 900, 1000, and 1300°C.

Task 3.4: Metallography of tensile-tested specimens after various heat treatments.

Task 3.5: Wire Making - Swaging, drawing, and annealing steps will be developed to draw the extruded rod into 0.0253-in.-diam wire.

Phase 4: Testing

In this phase, the wire produced at ORNL will be tested as heating elements at Hoskins.

The overall accomplishments during this CRADA are described below.

Completion of Objective

Between the work carried out at ORNL and at Hoskins, the objective of this CRADA has been met.

CRADA Benefits to DOE

The major benefit to DOE was the development of a new heating element alloy with significant potential savings in energy through its extended lifetime. Another benefit is the knowledge base developed at ORNL for requirements of melting and wire production under commercial production conditions.

Technical Discussion

Introduction

The FAPY is a Fe-16 at. % Al alloy of nominal composition in weight percent: aluminum – 8.46, chromium - 5.50, zirconium - 0.20, carbon - 0.03, molybdenum - 2.00, yttrium - 0.10, and iron - balance. The same chemistry was targeted for VIM heat 15512 at ORNL. The aluminum content of the alloy was chosen to maintain the alloy in the single phase (γ) without the formation of an ordered phase (DO_3). The alloy has good oxidation resistance up to 1000°C and has shown significantly superior performance as a heating element as compared to the commonly used nickel-based alloy Nichrome™. Being an α -alloy, it possesses nearly 20% elongation at room temperature in the fine-grain condition as opposed to approximately 10% for the ordered alloy. This report describes the work carried out towards developing procedures for melting, processing, and properties of FAPY alloy in support of producing heating element wire under commercial conditions at Hoskins.

Initial Wire Samples of FAPY Alloy for Heating Element Test

The first wire sample coil of FAPY alloy (ORNL heat 15512) measuring 0.0255 in. diam by 12 ft was shipped to Hoskins on January 14, 1994. The wire was produced by extruding a 15-lb VIM heat of the alloy. The hot-extruded bar was cold-swaged and wire-drawn with approximately 20% reduction per pass. The intermediate anneals as required were carried out at 800°C in air. The shipped wire was in the as-drawn condition from the last anneal that was given at 0.030 in. diam. This meant that the shipped wire had 40% cold work. The wire was to be evaluated by Hoskins for: (1) wire quality produced at ORNL and (2) for its performance under the thermal cycling test used for heating element evaluation.

The major objections by Hoskins to the ORNL-supplied wire were:

1. It was produced by VIM and all of Hoskins' current product are AIM.
2. Swaging was used in converting the hot-extruded rod to wire, and it is not a process used in the commercial wire production at Hoskins.
3. Uncertainty regarding applicability of the processing of small ingots at ORNL be applicable to the 8- by 8-in. tapered AIM ingots.

As a first step to answering Hoskins' questions, detailed processing steps were developed for 15-lb AIM and VIM heats at ORNL. Details of processing of these two heats are given in the next section.

Melting and Processing of 15-lb Heats of FAPY Alloy by AIM and VIM

One 15-lb FAPY heat each of AIM and VIM was made at ORNL using ZrO₂ crucibles. The target and actual analyses of each of the heats are compared in Table 1. The chemical analysis comparison shows the following:

1. Zirconium, which was added at 1.2 times the target in AIM, was recovered 100% and, therefore, does not need compensation.
2. Major elements for the AIM and VIM materials are essentially the same and match the target levels closely.
3. Carbon, nitrogen, and oxygen levels are higher for the AIM material. Although very low, the nitrogen and oxygen levels are nearly double for the AIM material versus the VIM material.
4. Yttrium was not detected in either of the melts.

Table 1. Comparison of chemical analysis of FAPY alloy melted in vacuum and air

Element	Weight percent		
	Target	VIM ^a	AIM ^b
C	0.03	0.032	0.040
S	--	0.002	0.003
Cr	5.5	5.30	5.27
Mo	2.0	2.09	2.07
Nb		<0.01	<0.01
Al	8.5	8.59	8.45
Zr	0.2	0.22	0.26 ^c
Fe	83.67	83.76	83.89
N ₂	--	0.003	0.008
O ₂	--	0.006	0.011
Y	0.10	ND ^d	ND ^d

^aVIM = vacuum-induction melted (Heat 16290).

^bAIM = air-induction melted (Heat 16292).

^cZirconium in air was charged as 1.2 × target.

^dNot detected.

The 2.85-in.-diam ingots of AIM and VIM heats were hot extruded at 1100°C through a 0.508-in.-diam die. The hot-extruded rods of both the AIM and VIM materials were subsequently warm- and cold-drawn to produce 0.024-in.-diam wire. The detailed drawing procedure for the AIM heat is shown in Table 2, and for the VIM heat in Table 3.