



Liquid phase separation in transition element high entropy alloys



A. Munitz ^{a,*}, M.J. Kaufman ^b, R. Abbaschian ^c

^a Nuclear Research Center-Negev, P.O. Box 9001, Beer-Sheva, 841900, Israel

^b Department of Metallurgical and Materials Eng, College of Applied Science and Engineering, Colorado School of Mines, Golden, CO, USA

^c College of Engineering, University of California, Riverside, CA 92521, USA

ARTICLE INFO

Article history:

Received 7 November 2016

Received in revised form

1 January 2017

Accepted 18 March 2017

Keywords:

High entropy alloys
Stable miscibility gap
Phase separation
Supercooling
Metastable

ABSTRACT

Non-consumable arc melting was used to produce buttons of transition metal (Co-Cu-Fe) high entropy alloys (HEAs) with additions of Al, Cr, Mn, Ni, V or Ti. High resolution scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) capabilities were used for microstructural characterization. It was found that certain HEAs exhibit stable liquid miscibility gaps causing separation into two liquids, namely a Cu-lean liquid, L1, and a Cu-rich liquid, L2. Since, in stable liquid phase separation (LPS) involving Cu, the Cu-rich L2 has a higher density and a lower melting temperature, it sinks to the bottom of the casting. Secondary melt separation was also observed and resulted in small spheres of the minority liquid embedded in the matrix of the first liquid. Additions of Co, Al, Ti, and Ni were found to lower the miscibility gap temperature (T_{MG}), while additions of Cr, V, and Nb were observed to raise T_{MG} and enhance stable LPS. Thermodynamic calculations were used to support the experimental results.

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1. Introduction

The existence of stable liquid phase separation (LPS) in high entropy alloys (HEAs) was reported by Hsu et al. [1] for AlAg-CoCrCuNi¹ and AlAgCoCrCuFeNi alloys and was confirmed by Munitz et al. [2]. It was found that cooling of these alloys during solidification resulted in LPS and produced an AlCoCrNi(Fe) rich liquid (L1) and a second liquid enriched in Ag and Cu (L2) [2]. Stable LPS is expected when adding Ag to an AlCoCrCuNi HEA since the binary Ag-Cr [3], Ag-Fe [4], Ag-Co [5], and Ag-Ni [6] phase diagrams all contain stable liquid miscibility gaps. The question remains, however, if the addition of other elements such as V to HEAs will favor stable LPS since only one of the binary diagrams with V (i.e., V-Cu [7]) contains a stable miscibility gap.

Stable LPS was also observed in CoCrCu_xFeMoNi when $x \geq 0.5$ [8] and in CoCrCuFe_{0.5}Ni and CoCrCuFeNi_{0.5} alloys [9]. On the other hand, Liu et al. [10] reported metastable LPS in CoCrCuFe_xNi (x between 1 and 2) alloys when a critical supercooling of 160, 190, and 293 K was reached for alloys with x equal 1, 1.5 and 2,

respectively. Wang et al. [11] reported metastable LPS in a CoCr-CuFeNi HEA when supercooled beyond 223 K. In spite of these findings, the majority of the recent research on HEAs has been concentrated on the relationship between the microstructure and mechanical properties of the HEAs [12–14] and on production routes to improve their mechanical properties [13–19].

Wang et al. [20] reported that adding 4 at% of either Mn, Co, Al, or Ni to a Cu-48 Fe alloy resulted in a reduction in the metastable miscibility gap temperature in the various pseudo-binary phase diagrams thereby suppressing the occurrence of metastable LPS. On the other hand, additions of Cr, V, Nb, Mo, Si, V or C to an equiatomic Cu-Fe alloy resulted in an increase in the metastable miscibility gap temperature; in some cases, the increase was sufficient to form a stable miscibility gap [9]. It was shown that the effects of Nb and C additions are, respectively, about two and twelve times higher than that of Cr [9]. It is of interest to determine the effects of adding or replacing elements such as Mn, V, Al, Nb and Ti in CoCrCuFeNi alloys on the T_{MG} and how this will affect the microstructure of these alloys during solidification. In addition, since the solidification path of an alloy separated into two liquid phases is considerably different and more complicated than that of a single phase supercooled liquid [22] it is of interest to investigate the solidification of supercooled liquids in these systems given that each liquid will experience its own supercooling, which can be considerably different from the bulk supercooling; therefore, the liquids will have different driving forces for solidification at any given

* Corresponding author.

E-mail address: munitzabr@gmail.com (A. Munitz).

¹ Throughout this paper, the compositions are listed as indicated here and indicate equiatomic amounts of the elements unless a subscript is used. For example, AlCoCrCu_{0.5}Ni corresponds to 26.3 at% of all of the elements except for Cu which is half that amount or 13.15 at%.

Table 1
Summary of nominal alloy compositions used in this study.

Alloy	Composition (At. Pct.)
Cu-Co	Cu-30Co
Co-Cu-Fe	Cu-15Co15Fe
CoCrCuFe	Co-25Cr-25Cu-25-Fe
CoCrCuFeNi	Co-20Cr-20-Cu-20Fe-20Ni
AlCoCrCuFeNi	Al-16.6Co-16.6Cr-16.6-Cu-16.6 at.%-Fe-20Ni
CoCrCuFeNiNb	Co-16.6Cr-16.6Cu-16.6Fe-16.6Ni-16.6Nb
CoCrCuFe	Co-25Cr-25Cu-25Fe
CoCrCuFeV	Co-20Cr-20Cu-20Fe-20V
Al0.5CoCrCuFeV	Al-18.2Co-18.2Cr-18.2Cu-18.2Fe-18.2V
AlCoCrCuFeV	Al-16.6Co-16.6Cr-16.6Cu-16.6Fe-16.6V
CoCrCuFeV	Co-20Cr-20Cu-20Fe-20V
CoCrCuFeTi0.5V	Co-18.2Cr-18.2Cu-18.2Fe-9.1Ti-18.2V
CoCrCuFeTiV	Co-16.6Cr-16.6Cu-16.6Fe-16.6Ti-16.6V
CoCuCrFeV0.5	Co-22.2Cu-22.2Cr-22.2Fe-11.1V
CoCrCuFeV	Co-20Cr-20Cu-20Fe-20V
CrCuFeMnV	Cr-20Cu-20Fe-20Mn-20V
CrCuMnTiV	Cr-20Cu-20Mn-20Ti-20V

temperature/undercooling. In addition, the effects of the latent heat released by the first liquid to solidify will influence the supercooling of the second liquid.

The purpose of this work was to achieve a better understanding of LPS in HEAs and the influence of alloying additions on the solidification structures. In particular, the effects of additions or substitutions of V, Al, Nb and Ti to a CoCrCuFe HEA on LPS and the resulting microstructures will be described.

2. Experimental procedure

A number of CoCrCuFeNi-X HEAs containing one or more of the following elements: V, Al, Nb and Ti (Table 1) was prepared by non-consumable arc melting in a Ti-gettered argon atmosphere. Elemental metal pieces (purity >99.5%) were utilized as the raw materials. All the elements were inserted into the Cu hearth with Cr or Cr + Fe being placed on top. The chamber was evacuated with a rotary pump to about 4–6 Pa at which point commercial high purity argon gas was purged into the chamber up to a pressure of 2 KPa, and the process was repeated. After arc ignition, a Ti getter was melted first, followed by melting of the alloying elements for about 30 s. The buttons were flipped over and the melting process was repeated 5 times to improve the chemical homogeneity. The dimensions of the buttons after melting were about 30 mm in diameter and a maximum height of about 9 mm. The buttons were

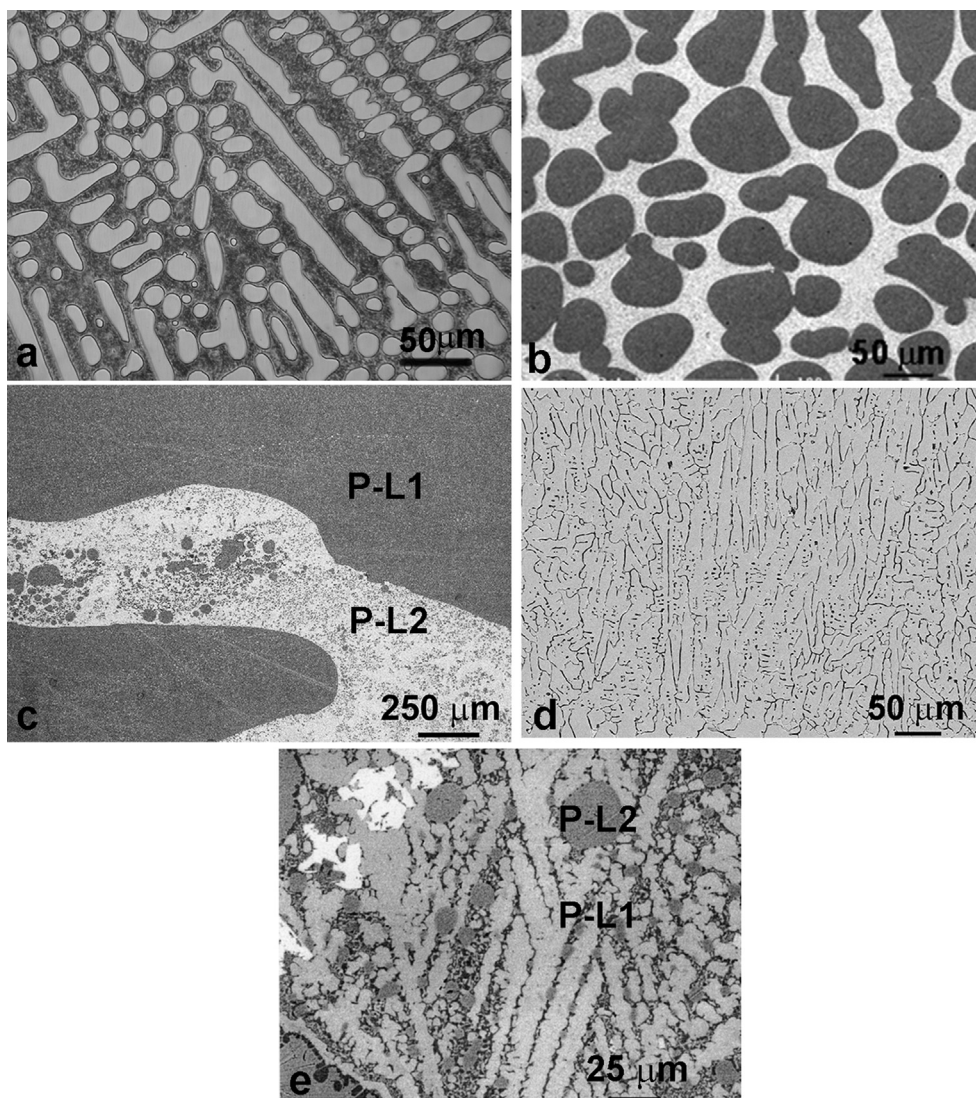


Fig. 1. Scanning electron micrograph (SEM) (a), optical photograph (b), and backscattered electron image (BEIs) (c–e) of the cross-sections of different alloy buttons: (a) Cu-30Co; (b) Cu-15Co-15Fe; (c) CoCrCuFe, (d) CoCrCuFeNi and (e) CoCrCuFeNiNb.

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