SEMESTER REPORT

Arfaoui Mohamed

Ph.D. thesis title: Structural characterization of multicomponent alloys and compounds in bulk and thin film form

Supervisor: Prof. György Radnóczi ,
Co-supervisor: Dr. Viktoria Kovács-Kis,
Place of research: Centre for Energy Research, Thin Film Physics Laboratory

Introduction

The main aims during the fourth semester were divided into tow part, finishing and submitting the manuscript which I have mentioned in the last previous semester report on in-situ TEM annealing from RT to 700°C (in 50°C steps/5 minutes) of thin CrFeCuNiCo HEA film, and the other issue was about preparing the poster for the ICAM international conference on advanced Materials which had already done in NICE from 26 to 30 of May. The poster results will be the concerned for the next publication.

The first part contains the pre-submitted mansusript, the second part will be concerned on the poster and we will end up with the attending lectures and exams

Part .1 Manuscript

I attached the manuscript in pdf word. the submission will be done within these days

Part 2 Poster

Abstract

High entropy alloy (HEA) thin films have attracted research interest because of simple structure, slow diffusion, good corrosion resistance and, rendering them a great potential as high temperature structural materials for different applications. Thus, it is a critical issue studying their structural stability during high temperature annealing. The aim of the present work is growing CrFeCoNiCu HEA thin films of 50 nm thickness andannealing them in-situ in the electron microscope for a better understanding and controlling the early stages of phase instability of as deposited films at temperatures up to 550 oC. The as deposited CrFeCoNiCu alloy films havesingle phase fcc structure and a grain size of about 10nm. No changes in their structureare detected up to 400oC. Separation of components is observed at 450 oC,where both the appearance of a bcc phase with a lattice parameter of 0.30 nm is registered as well as grain growth starts. Grains of 100 nm in size are forming in which the deviation from originally present equiatomic composition is measured by elemental mapping in STEM.Chemical separation and grain size growth as a function of time at 450 oC is presented

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Structural changes in CrFeCoNiCu high entropy thin films during in-situ annealing in TEM.

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Introduction

High entropy alloy (HEA) thin films have attracted research interest because of their simple structure, slow diffusion, and good corrosion resistance rendering them a great potential as high temperature structural materials for different applications. Thus, it is a critical issue studying their structural stability during high temperature annealing. The aim of the present work is to grow CrFeCoNiCu HEA thin films of 50 nm thickness and to study their structural changes by in situ TEM during annealing. The results contribute to a better understanding and controlling the early stages of phase instability of as deposited HEA films at temperatures up to 550 oC.

Experimental details

The equiatomic CrFeCoNiCu high entropy (HEA) films were deposited in a high vacuum system by direct current magnetron sputtering. The in-situ TEM heating at 450°C was performed with different annealing periods, followed by recording BF and SAED images from the same area of 1 µm in diameter at the end of each temperature step. Scanning transmission electron microscope (STEM), equipped with energy dispersive x-ray spectroscopy (EDS) was used to examine the crystal structure and the chemical composition of the new phases.

Results

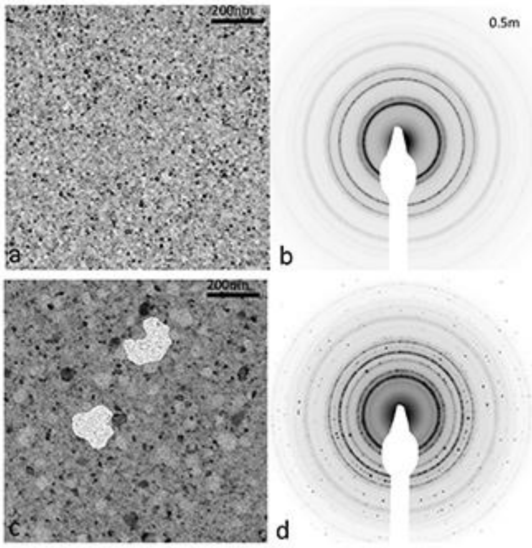


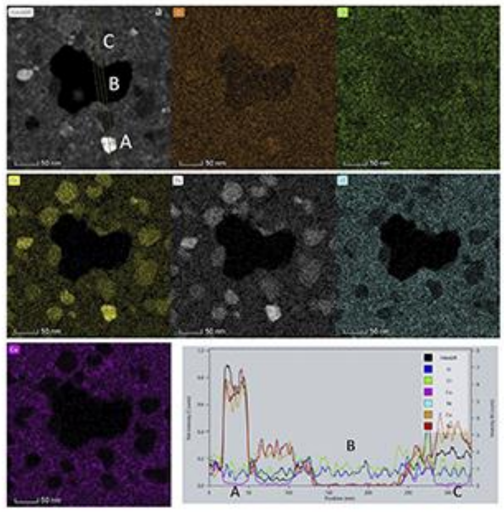
Fig1. BF and SAED pattern of the HEA film before (a,b) and after (c,d) annealing at 450°C for one hour.

As-deposited: The phase of the CrFeCoNiCu sample appears to be a single solid solution fcc phase with lattice parameter a(fcc)=0.360 nm. The mean grain size is about 7-10 nm.

Annealed at 450°C: Two phase structure, a new bcc phase is formed with lattice parameter a(bcc)=0.296 nm.

The host fcc phase, as well as the new bcc phase, undergoes grain growth at 450°C up to about 30-50 nm of grain size. Based on HRTEM (Figure 3) the following crystallographic relation can be established: d(111) of the fcc phase =d(110) of the bcc phase (0.208 nm).

Fig.2. Representatative HADAAF-STEM image of the annealed CoCrCuFeNi film annealed at 450 °C (a), EDS elemental maps show the distriubution of O, Cr, Cu, Fe, Co and Ni. The line profiles across areas A,B and C are shown.



Compositional changes:
chromium is immobile due to its bonding to oxygen on the surface, giving a CrO type oxide layer on the surface of the HEA film.
Rounded polycrystalline grains of 30-50 nm in diameter are either rich in Fe and Co, or in Ni.

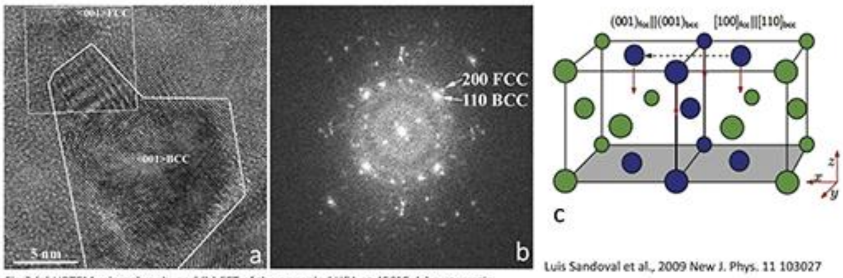


Fig.3 (a) HRTEM microghraph, and (b) FFT of the annealed HEA at 450°C, (c) systematic geometry of Bain orientational relationship.

Luis Sandoval et al., 2009 New J. Phys. 11 103027

An example of the observed special crystallographic orientation relationship between the fcc and bcc phases is shown in Figure 3. It corresponds to (100)bcc // (100)fcc and [001]fcc // [001]bcc, the so called Bain relation.

Summary

In the CrFeCoNiCu HEA film, annealed at 450°C, both the nanocrystalline fcc matrix and the newly formed bcc phase are homogeneously distributed on the nano-scale exhibiting the following crytalloghraphic relationship: d(110)bcc=d(111)fcc. Another orientational relationship, namely the Bain relation ((100)bcc //(100)fcc and [001]fcc//[001]bcc), characteristic for diffusionless transformation from fcc to bcc phase is observed. Redistribution of the alloying elements occurs. Grains enriched either in Fe and Co or in Ni appear. Cr becomes a continuous surface (protecting) layer in oxide form.

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Course and exams

Lattice deffec II / Dr. Gubicza Jenő , Exam 06/04/2019

Physical materials science I / Dr. Groma István , Exam 6/13/2019