Effect of Al Content on the Microstructure and Properties of As-Cast Al\textsubscript{x}CoCrCuFe Alloys

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Abstract
High-entropy alloys (HEAs) (or multi-principal component alloys) have been mentioned in different studies because they have good corrosion resistance and mechanical properties, but their biocidal ability is still little explored. The microstructural, magnetic, corrosion, and biocidal behavior of as-cast Al\textsubscript{x}CoCrCuFe high-entropy alloys with \( x = 0.5 \) and 0.9 were analyzed using scanning electron microscopy, energy-dispersive X-ray spectroscopy, magnetization measurements as a function of the applied field, X-ray diffraction patterns, electrochemical essays, and antimicrobial analyses. The results indicated that a higher concentration of Al reduces the saturation magnetization, the corrosion potential, as well as the survival time of the bacterium \textit{Escherichia coli} on the surface of the studied alloys. The dendritic microstructure was also refined with the increase in the concentration of Al and a higher number of Cu-rich precipitate was observed on their surface.

Graphical Abstract

Keywords  AlCoCrCuFe alloys · Biocidal behavior · High-entropy alloys · Microstructure
Introduction

High-entropy alloys (HEAs) (or multi-principal component alloys) are materials composed by five or more elements with similar molar fractions, that is, they do not have a principal component [1]. Due to the increase in their configurational entropy, HEAs can present a lower number of phases than that expected for traditional alloys. This leads to interactions that normally would not exist among the alloying elements, modifying the properties of the material. The promising characteristics of these materials are linked to their mechanical properties and corrosion resistance [1]. Some researchers have also shown the HEAs ability to resist microorganism-induced corrosion (MIC) [2–4]. This type of corrosive damage generates significant losses to industries that operate in susceptible environments to microorganism growth [5]. It is still necessary to highlight the importance of research focused on antimicrobial materials due to the recent SARS-CoV2 pandemic [6, 7], since applying or making surfaces of such materials can reduce transmission via fomites [8]. Metallic copper, its ions in solution, as well as copper-based alloys have been, empirically, used for medical applications by ancient people in many regions around the world [7]. The survival of pathogenic microorganisms on surfaces of metallic Cu and Cu-based alloys was assessed in different studies [7–11], but in most cases, the Cu concentration was higher than 60 wt.% or around it. Li et al. [12] studied the antiviral properties of the CuFeCrCoNi and Al0.4CuFeCrCoNi high-entropy alloys with lower Cu content, showing over 99.99% of efficiency under influenza H1N1 virus and enterovirus after 24 h in contact. These results were related to the interaction of copper ions, hydroxyl radicals, and microorganisms, revealing new possibilities for the application of high-entropy alloys as biocidal materials.

Considering the evaluated compositions, no information was found in the literature about the biocidal characteristics of Al0.9CoCrCuFe multi-principal element alloys. Ivchenko reported that the microstructure of as-cast AlCrCoCuFeNi alloy is made up of dendrites that can be decomposed into many phases with different morphologies and chemical compositions. This alloy was also susceptible to heat treatment [13]. Jones et al. evaluated the Al0.5CoCrCuFeNi multi-principal element alloy and they verified that the configurational complexity was not enough to stabilize a solid solution phase against enthalpic effects [14]. Another report by Jones et al. [15] verified that the configurational entropy was not dominant over enthalpy for the contribution of Gibbs energy in the Al0.5CoCrCuFeNi alloy. Data from the literature for the Al0.5CoCrCuFeNi multi-principal element alloy also exhibited that the B2 phase, with solvus temperature around 1248 K, is quickly formed in this composition [16]. Furthermore, precipitates of a Cu-rich phase were formed in the dendrites, while a CrCoFe-rich phase was precipitated in the interdendritic region of the Al0.5CrCoCuFeNi alloy [17]. Thus, the possibility of Cu-rich precipitate formation with the variation of the Al concentration appears as an efficient tool to improve the biocidal capacity of high-entropy alloys containing Cu, due to its higher availability to microorganisms.

In this sense, the present study aimed to evaluate the microstructural, magnetic, and biocidal behavior of as-cast Al1.0CoCrCuFe high-entropy alloys with copper content around 20 at.% (25 wt.%), as well as the effects of change in Al concentration on these properties, including the formation of Cu-rich precipitates.

Materials and Methods

The Al0.3CoCrCuFe and Al0.9CoCrCuFe multi-principal component alloys were prepared from materials with a purity higher than 99.5%, using an electric arc furnace. The produced ingots were cut into pieces suitable for obtaining images by optical (OM) and scanning electron microscopy (SEM), as well as for biological assays. Details about the adopted procedures for melting, cutting, and preparing samples can be found in reference [18]. An Olympus optical microscope and a Quanta 650FEG scanning electron microscope coupled with energy-dispersive X-ray spectroscopy (EDS) and with a backscattered electrons detector were used for this study. The area percentage of the interdendritic region was quantified using the free software ImageJ. A D8 Discover diffractometer with Cu Kα radiation was utilized to define the X-ray diffraction patterns (XRD) from bulk samples. A PPMS 9 Evercool Quantum Design was used to monitor the magnetization change with the magnetic field (M vs. H) at room temperature in samples with a mass of about 2 mg. An Autolab PGSTAT302N Metrohm potentiostat/galvanostat, with a conventional three electrodes cell containing the Ag/AgCl reference electrode, platinum wire as a counter-electrode, and the sample as a working electrode, monitored the potentiodynamic polarization measurements from −350 mV versus open circuit potential (OCP) up to 1000 mV versus reference electrode with a scan rate of 1 mV s−1. Previously, the OCP was recorded for 24 h. These assays were performed in a solution of NaCl 1.0 mol L−1 with a pH = 6.3 at room temperature. The experiments were carried out in quadruplicate and the solution was not deaerated. Antimicrobial tests or biocide activity of the alloys were determined by the adaptation of protocol described by ISO 22196/2011, based on the recovery of viable cells after direct contact between the microorganism and the surface.
of the alloy. *Escherichia coli* (ATCC 8739) was applied as a model pathogen for the biocide experiments. The bacterial culture was cultured in Luria Bertani medium (Invitrogen™) at 308 K for 16 h, followed by its inoculation as a microbial suspension (10^6), on samples of Al_{0.5}CoCrCuFe alloys. After time 0 h (immediately after the contact) and 24 h, at 308 K, the experiment was monitored to verify the recovery of viable cells.

**Results and Discussion**

Figure 1 shows the scanning electron and optical micrographs obtained for the Al_{0.5}CoCrCuFe and Al_{0.9}CoCrCuFe alloys. From optical micrographs in Fig. 1a and c, it was possible to notice that both alloys presented dendritic microstructure, with well-defined interdendritic regions. Dendrites were formed during the cooling, mainly by the fact that on fast cooling a part of the liquid can be undercooled and rapidly solidify from an initial nucleus, growing with high surface energy. This is common in as-cast material and can lead to a difference in concentration that can form two different liquids that solidify at slightly different temperatures or it can lead to the formation of an intermetallic compound in the interdendritic region.

In OM images (Fig. 1a and c) it was possible to see that the increase in aluminum content seems to refine the dendritic microstructure and increase its fraction (58% of the area in Fig. 1a and 64% of the area in Fig. 1c). This was an interesting result since light elements like Al tend to segregate and disturb the simple solid solution formed in HEAs. In this case, the increment in Al seems to stabilize the solid solution. The scanning electron micrographs in Fig. 1b and d showed details of the interdendritic and dendritic microstructure. In SEM images of Fig. 1b and d, it was possible to notice the presence of small precipitates on the dendritic region of the alloys and that their amount was increased with Al content. EDS data indicated that they are Cu-rich precipitates, with reduced Al fraction and other minor alloying elements. This was an interesting fact because even with the increase in precipitates amount with Al content, the dendrites appear to be thicker, indicating that possibly this

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**Fig. 1** OM and SEM images obtained for the etched alloys: (a, b) Al_{0.5}CoCrCuFe and (c, d) Al_{0.9}CoCrCuFe

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precipitation reaction occurred after the alloy solidification, and also it did not interfere with dendrites growth in the studied alloys.

This is an interesting finding since Cu and Al have a certain chemical affinity. To better understand we can resort to the mixing enthalpy of the elements in the alloys, which is related to the chemical interaction between these elements. As one can notice in Table 1, Cu and Al show a chemical affinity ($\Delta H_{\text{mix}} < 0$), in a matter of fact, Cu only presents a chemical affinity to Al. So, at first glance, it is intriguing that the increase in Al increases the amount of segregated Cu.

However, in a deep evaluation, we can notice that Al presents a higher interaction with Cr ($-10$ kJ/mol), Fe ($-11$ kJ/mol), and Co ($-19$ kJ/mol) than with Cu ($-1$ kJ/mol). This leads to the fact that when the Al fraction is increased, it is easier for the elements to bond with the Al and reject the Cu atoms.

Considering the Gibbs mixing energy (given in Eq. 1) as a spontaneity factor for the formation of a solid solution it is possible to notice that as the mixing enthalpy is constant, the contribution of entropy depends on the temperature. So, at high temperatures the entropy contribution can be significant and stabilize the Cu in the dendrites; however, as the temperature decreases the enthalpy contribution is prevalent and the Cu is no longer stable in the dendrite, precipitating onto those.

$$\Delta G_{\text{mix}} = \Delta H_{\text{mix}} - T\Delta S_{\text{mix}}$$

We can further expand Eq. 1 by rewriting the entropy using the Boltzmann equation and the enthalpy using the Miedema model, as given in Eqs. 2 and 3.

$$\Delta S_{\text{mix}} = -R \sum_{i=1}^{n} e_i \ln(c_i)$$

$$\Delta H_{\text{mix}} = \sum_{i=1}^{n} \sum_{j \neq i} 4\Delta H_{\text{mix}}^{ij} c_i c_j$$

The calculated enthalpy and entropy for the studied alloys are presented in Table 2. As expected, the addition of Al stabilizes the solution decreasing the mixing enthalpy.

| Al atomic ratio | $\Delta H_{\text{mix}}$ | $\Delta S_{\text{mix}}$ |
|----------------|-------------------------|--------------------------|
| at./at        | kJ/mol                  | J/mol K R                |
| 0.5           | 0.22                    | 13.15 1.58               |
| 0.9           | -0.50                   | 13.37 1.61               |

In order to evaluate the compositional difference in observed microstructures, some SEM images were obtained using backscattered electrons. These ones can be seen in Fig. 2. It was possible to notice that the dendritic and interdendritic regions were indeed chemically different and precipitates on the dendritic region were observed, as also seen in Fig. 1b and d. To assess this different composition, it was conducted point analyses by energy-dispersive X-ray spectroscopy (EDS) in the regions denoted by the numbers 1 and 2 in Fig. 2. The resulting spectra can be seen in Fig. 3.

From the spectra presented in Fig. 3, it is possible to obtain the semiquantitative determination of the alloying elements, as shown in Table 3. In both alloys, the interdendritic (region 1) is mainly composed by Cu and Al, while the dendritic (region 2) is composed mainly of Fe, Co, and Cr. However, with the increase in Al content, the fraction of it in region 2 is raised more than in region 1. This difference can be seen in the EDS composition maps exhibited in Fig. 4.

In order to understand the structural information behind the discussed chemical difference, X-ray diffraction patterns for the studied alloys were obtained and can be seen in Fig. 5a. It was possible to notice that both alloys presented similar diffraction peaks; however slight changes in position and intensity were also verified, as shown in the inserted graph in Fig. 5a. These diffraction peaks were consistent with two solid solutions, one of them the face-centered disordered solid solution with Space Group $Fm\overline{3}m$ (ICSD 606,883 [20]), henceforth called SS$_{fcc}$, and the other a body-centered disordered solid solution with Space Group $Im\overline{3}m$ (ICSD 102,751 [20]), henceforth called SS$_{bcc}$.

The presence of two phases in alloys with five components indicates a dominant effect of entropic factors concerning enthalpic ones. These findings were in agreement with the dendritic region, SS$_{bcc}$, and interdendritic regions SS$_{fcc}$ shown in Figs. 1, 2, and 4. It is also important to verify that the peak intensity from SS$_{bcc}$ increased when Al was added to the alloy. This is expected since a reduction has been seen in the interdendritic region (see Fig. 1a and c). The second effect of Al addition to the alloy is the Cu and Al content increase and the Co, Cr, and Fe decrease in the dendritic region. This effect was compatible with the increase in the quantity of Cu-rich precipitates observed in the SEM images in Fig. 1b and d.

The main alloying elements forming the dendritic region of materials are Fe, Co, and Cr. These elements are often
associated with the appearance of magnetic properties in different types of materials. Thus, if the variation proposed above is consistent, a change in the saturation magnetization of the material can be verified with the increase in the Al content. Thus, to confirm the description presented magnetization measurements with applied field were performed at 300 K, as seen in Fig. 5b.

In Fig. 5b it was possible to notice that the saturation magnetization decreased with the increase in Al content. This result is in agreement with the decrease in the relative fraction of Fe, Co, and Cr in the dendritic region and suggests a direct relationship of these elements with the magnetic effect observed in the alloys. Considering that the dendritic region is associated with the ferromagnetic
behavior in these alloys, the reduction in the saturation magnetization ($M_s$) when Al is added may be related to the substitution of Co, Cr, and Fe by Cu and Al atoms. Therefore, even with a higher relative fraction of Cu in the dendritic region with increasing Al content, this copper is less available than that segregated in the interdendritic region.

Literature data [21] already show that the presence of Cu in multi-principal component alloys can impair their corrosion resistance. Therefore, the difference in the relative distribution of Cu in the metallic matrix due to the increase in Al can bring different results for the corrosion resistance of the analyzed alloys.

In order to evaluate the corrosion resistance of the alloys, potentiodynamic polarization curves were acquired for both the studied materials. Figure 6a shows the collected curves using a 1.0 mol L$^{-1}$ NaCl(aq) solution, immediately after monitoring the open-circuit potential for 24 h. In these curves, it is possible to notice the corrosion potential ($E_{corr}$) and the current density ($i_{corr}$) are: $−185$ mV/3.59 μA cm$^{-2}$ and $−225$ mV/0.412 μA cm$^{-2}$ for the Al$_{0.5}$CoCrCuFe and Al$_{0.9}$CoCrCuFe alloys, respectively. These values
were obtained employing the Tafel extrapolation [22]. It is important to highlight that $E_{\text{corr}}$ and $i_{\text{corr}}$ values for the Al$_{0.9}$CoCrCuFe alloy are similar to those verified by Qiu et al.[23] in an equiatomic AlCoCuCrFe alloy.

It is possible to notice that the corrosion potential observed for the Al$_{0.9}$CoCrCuFe alloy was decreased, indicating a more cathodic potential and a higher susceptibility to corrosion when compared to the Al$_{0.5}$CoCrCuFe alloy. Besides that, the extension of the pseudo-passive region for the Al$_{0.9}$CoCrCuFe alloy was almost the same (96 mV vs 100 mV for the Al$_{0.5}$CoCrCuFe alloy). This region can be related to the formation of porous or poor protective oxide [24]. It is important to clarify that the corrosion tests were carried out just after the sanding of the surface of the sample; therefore, there was no time to form an appreciable oxide layer. So, the pseudo-passivity can be attributed to the incomplete formation of a protective oxide layer, with a similar protective character for both alloys. In addition, the displacement to lower values of current density observed for the Al$_{0.9}$CoCrCuFe alloy suggests a lower corrosion rate when compared to the Al$_{0.5}$CoCrCuFe Al alloy [25].

The oxide film will likely be richer in Al in the alloy that contains a higher amount of this element. As proposed, a more protective oxide in the alloy with the highest amount of Al was expected, as provided by Pourbaix diagrams [26]. It reveals the passivity promoted by Al combined with oxygen has a more extensive region than that offered by Cr or other elements present in the investigated alloys. Besides that, the passivity promoted by Al and Cr oxides is widely known [27, 28]. Then, although the $E_{\text{corr}}$ potential is lower for the Al$_{0.9}$CoCrCuFe alloy, the lower corrosion rate inferred by $i_{\text{corr}}$ suggests a higher protective character compared to the Al$_{0.5}$CoCrCuFe alloy. However, once the protective layer for the Al$_{0.9}$CoCrCuFe alloy is broken the corrosion takes place and the alloy becomes more susceptible to corrosion when compared to the alloy with lower Al content. This was coherent with electrochemical data collected for a similar system, such as AlCoCrFeNi alloy [29].

It is known that the diffusion of matrix atoms also occurs through the passive layer formed on the surface of metallic alloys and that in the presence of a Cu-rich region and another with Fe, Co, and Cr, the copper can oxidize preferentially [21]. Thus, the effect of Cu on bacteria in contact with the surface of the material may be dependent on the physicochemical and microstructural characteristics of the system under analysis.

As a form to evaluate the biocidal properties of these alloys, since Cu is commonly described by literature about this ability, the materials were analyzed regarding their microbicide activity under the bacterium *E. coli*, an important clinical pathogen and fecal contamination indicator for many processes.

In this sense, after evaluations of the microbial growth immediately after contact (0 h) and 24 h later, it was possible to verify the efficiency of the alloys as a biocide. Both polished and unpolished sides in alloys were exposed to a strain of *Escherichia coli* ATCC 8739. The results obtained for the polished and unpolished surfaces (Fig. 6b) showed the absence of bacterial growth on the surface of the samples after 24 h of contact. Figure 6b shows the behavior observed at 0 h. It was possible to notice that the unpolished samples of the Al$_{0.5}$CoCrCuFe alloy presented a higher colony count than the Al$_{0.9}$CoCrCuFe alloy at
time 0 h in the dilution factor of 0.1. The relative behavior of unpolished surfaces was the same at lower dilution factors for the two alloys.

Now, considering the polished sides of the alloys and the dilution factor of 0.1, it can be seen that after the immediate contact of bacteria with the alloys, there was no significant change in its development. However, when the dilution factor was decreased to 0.01, there was a significant reduction in the bacterial colonies, but in the same proportion in both alloys. For the dilution factor of 0.001, it can be verified that the number of bacteria colonies in the Al0.5CoCuCrFe alloy was decreased, while in the Al0.9CoCuCrFe alloy it reached zero (0). Thus, considering the polished and unpolished surfaces of the two alloys, the results indicated that the Al0.9CoCuCrFe alloy presents a better performance to inactivate the bacterial pathogen on its surface.

It is known that the inactivation of *Escherichia coli* on Cu-based alloy surface is mainly due to the effect of the Cu+/Cu2+ ion released during the oxidation process of the material [7–12]. As the multi-principal element alloys studied here are composed of five elements, we cannot reason that only copper is being oxidized, but the biocidal effect observed must be due mainly to the ions of this element [12]. Although the alloys have a high number of components, the experimental data obtained indicated the formation of a Cu-rich phase at the interdendritic region while the dendritic region is rich in Cr, Fe, Co, and Al (Fig. 4). It is known that this configuration allows the arising of galvanic effects between the dendritic and interdendritic regions. As the relative fraction of Cr in the dendrites is greater than 15at.%, their corrosion resistance is higher, making the Cu-rich region the anode of this galvanic cell. This allows the preferential corrosion of the Cu-rich solution and the formation of copper ions [21]. This mechanism may be responsible for providing copper ions for the inactivation of bacteria on the surface of the alloys. In addition, it has been reported that the survival of *Escherichia coli* began to decline when the copper ion concentration reached 400 mmol L⁻¹ and came to zero when the Cu concentration reached 600 mmol L⁻¹ [30, 31]. Therefore, the results seem to be related to the microstructure and corrosion behavior of the materials. The images in Fig. 4 showed that, regardless of the Al concentration, copper was deposited preferentially in the interdendritic region of the alloys, but in the Al0.9CoCrCuFe alloy this field seems to be reduced while a higher quantity of Cu-rich precipitates was verified in the dendritic region, as seen in Fig. 2b. Polarization tests showed that the alloy with the highest concentration of Al has a less noble corrosion potential, but its corrosion current density was also lower. This suggests that the observed biocidal effects may be closely related to the corrosion rate of the material and the preferential oxidation of copper. The Al0.9CoCrCuFe alloy exhibited the best results in action against *Escherichia coli*. It is important to mention that the corrosion current density is directly proportional to the corrosion rate of the material.

The fact that the unpolished alloys showed more interesting results in higher concentrations of bacteria may be due to the contribution of the metallic matrix, surface oxide layer, and corrosion rate. In the case of polished samples, the most significant contribution should be from the metallic matrix. The variation observed as a function of the bacteria concentration was due to the Cu+/Cu²⁺ ions concentration available to interact with the bacteria. As the time of contact with the metallic matrix was reduced, a lower content of Cu+/Cu²⁺ ions was produced when compared to the bacteria concentration, and only the number of bacteria proportional to this concentration of Cu+/Cu²⁺ ions can be inactivated at 0 h. This is remarkable in the unpolished material because the passive layer of the Al0.9CoCuCrFe alloy is a little more protective and the copper is better distributed throughout the matrix, which could also interfere with the availability of Cu ions. Hence, there is a relationship between the biocidal ability of the studied alloys and their microstructure and corrosion behavior.

**Conclusions**

The increase in the concentration of Al promoted the refinement of the dendrites and the interdendritic regions of the Al0.5CoCrCuFe and Al0.9CoCrCuFe alloys. Both studied alloys exhibited precipitates on the dendritic region that were related to an fcc solid solution, the same phase found in the interdendritic region. The alloy with higher Al content presented lower saturation magnetization and corrosion potential, but also exhibited a lower corrosion current density. These results were related to the biocidal ability of the alloys and the material with lower corrosion current density showed good performance under the bacterial strain *Escherichia coli* ATCC 8739.

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**Conflict of interest** On behalf of all authors, the corresponding author states that there is no conflict of interest.

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