

## SURFACE AND INTERFACIAL PROPERTIES OF Fe-C-O-Cr ALLOYS IN CONTACT WITH ALUMINA

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### Abstract

*In this paper, temperature and concentration dependencies on density and surface tension of liquid Fe-C-O-Cr alloys (1.93 - 4.80 wt.% Cr) were investigated in high-temperature resistance observation furnace by a sessile drop method during heating from liquidus temperature to 1600 °C. The interfacial characteristics (interfacial tension, wetting angle, work of adhesion, and spreading coefficient) of liquid alloy/alumina system were also determined depending on temperature. The effect of temperature and chromium content on surface and interfacial properties was proven in case of all examined alloys. Based on the fact that the content of surface-active elements such as oxygen (up to 195 ppm) and sulfur (up to 545 ppm) was higher, the influence of activities of both mentioned elements on surface tension of alloy samples was assessed. Particular attention was paid to the dependence of the surface tension temperature coefficient on oxygen and sulfur activity.*

**Keywords:** Fe-C-O-Cr alloys; Surface and interfacial tension; Density; Wetting angle; Effect of chromium; Sessile drop method

### 1. Introduction

During numerous metallurgical processes, particularly casting and refining, the metallic melt is in contact with a solid ceramic refractory material. At the interface between these systems, the interactions influencing physicochemical properties of the final product occur at high temperatures. Characterization of the ceramic material surface by determining the contact angle of metal drop resting on solid substrate contributes to the understanding of physicochemical processes at the phase interface and thereby optimizing the metal product. It can be assumed that non-wettable surfaces are more resistant to the melt exposure, i.e. during the production process these surfaces will be more inert to chemical and mechanical action.

The experimental study of the surface and interfacial properties of liquid multicomponent metals in contact with a ceramic material is demanding, especially, due to the possible high reactivity of metallic melts, chemical heterogeneity, roughness and porosity of the substrate surface, and sensitivity of surface tension to impurities. Besides, the determination of surface tension and the characterization of the phase interface (metallic melt/ceramic) is also intricate due to discrepancies in methodology and inconsistency in experimental procedures [1].

In principle, the surface tension or surface energy

arises from a phenomenon that atoms near a free surface have partially empty coordination shells and therefore they are at higher energy states than the atoms in the bulk of the solution. In other words, it is a surface physical force which shows that the atoms in the liquid bulk pull their neighbours in all directions in zero net force, contrary to the surface atoms experiencing a net inward force from atoms below. Nevertheless, in terms of a multicomponent alloy, atoms whose energy state is affected least by the surface are segregated to the liquid surface region. Chromium is among metals which segregates onto an alloy surface. Its energy changes concerning segregation are relatively negligible compared to strongly surface-active elements like sulfur and oxygen. Therefore, it influences the surface tension to a significantly smaller extent [2-6].

The effect of chromium has already been investigated by several researchers. Treťyakova et al. investigated the dependence of surface tension on chromium content (up to 1 wt.% Cr) in Fe-Cr-O systems. In this case, the minimum surface tension occurred when the melt was the most microheterogeneous. The presence of chromium increased surface tension due to its ability to penetrate between the clusters, which made the melt more uniform [7]. The influence of chromium on surface tension was also investigated by Mukai and Li [8, 9].

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They found that chromium slightly increased surface tension. Further, the chromium possessed a strong affinity to oxygen and facilitated its adsorption. Therefore, at higher concentrations (above 10 wt.%) oxygen lowered the surface tension of Fe-Cr-O systems [10].

It is assumed that for most liquid metals, alloys and steels, the temperature coefficient of surface tension is negative [11]. Nevertheless, especially for systems having considerable positive free energies, it can be positive [12]. Usually, the positive surface coefficient of surface tension is associated with surface segregation and higher values of the excess Gibbs energy [13]. Li noted that the coefficient of Fe-16 mass%Cr-S alloys increased with increasing sulfur content and was positive when sulfur content reached over 20 mass ppm [8]. Further, the effect of steel composition on temperature coefficient was investigated on several commercial 4-series ferritic stainless steels. It was found that it changed from negative to positive at a soluble sulfur content above 30 mass ppm in the steel [14]. Similar conclusions concerning sulfur influence were documented in literature [15, 16]. The extensive research on this issue was provided by Brooks, who examined more than 40 austenitic and ferritic steels and observed that for those with high sulfur content, the coefficient was positive [17]. Measurements of surface tension of low alloyed AISI 4142, Fe-Cr-Ni stainless steel AISI, and high-manganese Fe-Cr-Mn-Ni steel containing sulfur (> 50 ppm) revealed that temperature coefficient of surface tension was positive for all investigated alloys. It was suggested that the free surface of a liquid droplet was covered by a monolayer of surface-active elements, e.g. sulfur, causing a reduction of surface tension, whereas, when the temperature increased, sulfur was desorbed into the bulk of the liquid metal, and the surface tension rose [3, 18, 19].

This work is focused on the experimental study of surface and interfacial quantities regarding Fe-C-O-Cr alloys in contact with alumina substrate depending on temperature and change in chemical composition. Moreover, the influence of chromium and surface-active elements (oxygen and sulfur) on surface tension has been determined.

**Table 1.** Chemical composition of examined alloys

Sample	Cr	C	Mn	W	Co	O	S
	wt.% / at.%					ppmw / ppma	
1	1.925/ 2.037	0.345/ 1.581	0.049/ 0.049	0.015/ 0.004	0.020/ 0.019	187/ 643	521/ 894
2	2.970/ 3.141	0.342/ 1.566	0.050/ 0.050	0.024/ 0.007	0.020/ 0.019	195/ 670	522/ 895
3	3.772/ 3.988	0.335/ 1.533	0.053/ 0.053	0.028/ 0.008	0.014/ 0.013	167/ 574	545/ 934
4	4.796/ 5.071	0.340/ 1.556	0.042/ 0.042	0.044/ 0.013	0.010/ 0.009	15/ 52	62/ 106

The content of other elements (Ni, Si, Ti, Mo, P, Al, Cu, and Zr) present in alloys was less than 0.005 wt.%, and the rest represents iron.

## 2. Experimental

### 2.1 Samples preparation

Four alloys (samples 1-4) were selected for studying the surface and interface properties by a sessile drop method, a steady and accurate method for surface tension measurement at high temperatures having experimental error lower than  $\pm 2\%$ , as noted in [20]. These samples were prepared from pure metals (Fe and Cr, purity 99.99%), carbon (purity 99.99%), and Fe<sub>2</sub>O<sub>3</sub> tablets (purity 99.999%) by vacuum induction melting in furnace Leybold Heraeus at the working site. The melt was cast into the vertically oriented mould to obtain 3 kg ingots from which rods of a diameter 5 mm were made. Their chemical composition was determined by GDA 750 HP optical emission spectrometer (GDOES) as shown in Table 1. Carbon, oxygen, and sulfur contents were obtained by combustion analysers Eltra 2000 CS and Eltra 2000 ONH.

Prior to the experiment, cylindrical alloy samples (5 mm diameter x 5 mm height) were thoroughly mechanically cleaned to remove surface oxides. For the purposes of the experiment, alumina plates (99.8% Al<sub>2</sub>O<sub>3</sub>) were annealed at 1150 °C for 6 hours, and their surface was cleaned by acetone immediately preceding the measurements.

### 2.2 Experimental procedure

Experimental determination of density, surface tension, and wetting angle was carried out using the sessile drop method in the high-temperature observation furnace CLASIC (Figure 1) in the temperature range from the melting point of the alloy sample to 1600 °C. The prepared sample (alloy/Al<sub>2</sub>O<sub>3</sub> substrate) was placed in the furnace tube which was then hermetically sealed with a setup of approximately 0.1 Pa, and purged with argon of high purity (> 99.9999%). The latter two steps were repeated. The heating rate and maximum temperature were set to 5 °C min<sup>-1</sup>, and 1600 °C. The temperature was measured with a Pt-13% Rh/Pt thermocouple close to the sample. To prevent subsequent oxidation of the sample, which substantially affects surface

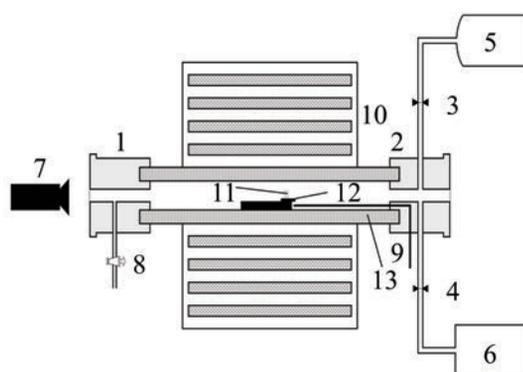


tension and temperature coefficient, as reported by Yuan [21], all measurements were carried out in an inert atmosphere of argon. Each sample was measured four times. The images of drop forming during a heating ramp were taken by a CANON EOS550D and then saved in a PC. Then, they were evaluated by the Laplace - Young equation, describing the equilibrium pressure at the interface. The evaluation was performed by in house software involving ADSA (Advanced axisymmetric drop shape analysis) method [22, 23]. This method determines parameters such as wetting angle, surface tension, density, etc.

The interaction of alloy samples with alumina substrate after the experiment was investigated by scanning electron microscope (SEM) JEOL 6490LV. The X-ray microanalysis of microstructural particles and their chemical composition was carried out by EDS (energy dispersive X-ray spectroscopy) analyzer INCA in the mode of backscattered electrons (BSE). The device setup was: thermo-emission cathode LaB6, voltage 20 kV, resolution of 3.0 nm, vacuum  $2.5 \cdot 10^{-6}$  Pa.

### 3. Results and discussion

Figure 2 depicts measured density data plotted against temperature for all investigated alloy samples. The temperature dependencies on density were investigated in the temperature interval from alloy melting point to 1600 °C during which a linear decline in density with increasing temperature was observed. The density further depended on the chromium content. At 1600 °C, the highest value ( $7.04 \text{ g} \cdot \text{cm}^{-3}$ ) was obtained for Sample 1 containing 1.925 wt.% Cr and the lowest ( $6.79 \text{ g} \cdot \text{cm}^{-3}$ ) for Sample 4 holding 4.80 wt.% Cr. In summary, the density decreased with higher chromium content, which was consistent with



**Figure 1.** The scheme of high-temperature observation furnace CLASIC 1) front flange, 2) rear flange, 3) gas inlet, 4) gas outlet, 5) high-pressure argon gas cylinder, 6) vacuum pump, 7) CCD camera, 8) high-vacuum stopcock, 9) type B thermocouple, 10) heating elements, 11) sample, 12) alumina substrate, 13) alumina tube

the findings published in articles [14, 19].

Parameters listed in Table 2 were obtained through the least square fitting of measured data to Equation (1) [19]

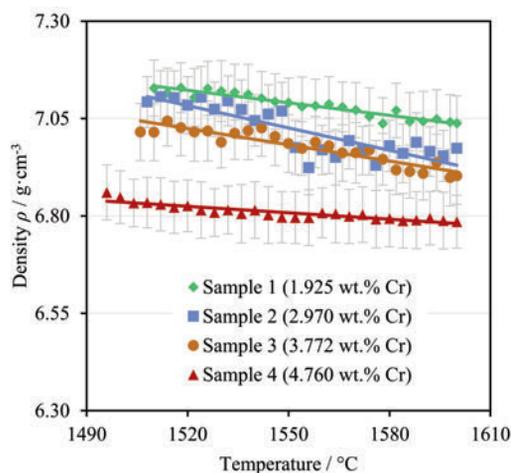
$$\rho(T) = \rho_{ref} + \frac{d\rho}{dT} \cdot (T - T_{ref}) \quad (1)$$

where  $\rho_{ref}$  is density ( $\text{g} \cdot \text{cm}^{-3}$ ) at reference temperature (liquidus temperature)  $T_{ref}$  (°C), and  $d\rho/dT$  ( $\text{g} \cdot \text{cm}^{-3} \cdot \text{C}^{-1}$ ) is the temperature coefficient of the density.

Experimental densities were compared with those calculated by Thermo-Calc software operating with the TCFE8 Steel/Fe-Alloy database. The database is applicable for different grades of Fe steels and alloys with the recommended content of specific alloy elements. Figure 3 presents a comparison of both densities, whereas a relative error was not larger than 3.5 %.

The temperature dependence on surface tension followed an upward trend for all examined alloys, as can be seen in Figure 4.

In all cases, the temperature coefficient of surface tension  $d\sigma/dT$  was positive and dependent on the activity of sulfur and oxygen (Figure 5). This phenomenon can be explained by the Gibbs adsorption



**Figure 2.** Density values plotted against temperature; temperature range between liquidus temperature and 1600 °C (error bars denote standard error)

**Table 2.** Calculated parameters of linear dependence of density on temperature for alloy samples 1-4

Sample	$T_{ref}$ / °C	$\rho_{ref}$ / g cm <sup>-3</sup>	$d\rho/dT$ / g cm <sup>-3</sup> °C <sup>-1</sup>	$\Delta T$ / °C
1	1510	7.13	$-1.08 \times 10^{-3}$	1510-1600
2	1508	7.09	$-1.91 \times 10^{-3}$	1508-1600
3	1506	7.02	$-1.39 \times 10^{-3}$	1506-1600
4	1496	6.86	$-0.54 \times 10^{-3}$	1496-1600



isotherm which assumes that the free surface of a metal drop is covered by a monolayer of surface-active elements like oxygen and sulfur tending to lower surface tension. During heating, these elements are desorbed into the bulk and, therefore, surface tension rises [19]. Such behavior was observed for samples 1-3 where the sulfur content was the highest, unlike for sample 4 which had the lowest content of surface-active elements (sulfur and oxygen).

The linear Equation (2) was fitted to the measured data of surface tension according to [19]

$$\sigma(T) = \sigma_{ref} + \frac{d\sigma}{dT} \cdot (T - T_{ref}) \quad (2)$$

where  $\sigma_{ref}$  is surface tension ( $\text{mN m}^{-1}$ ) at reference temperature (liquidus temperature)  $T_{ref}$  ( $^{\circ}\text{C}$ ), and  $d\sigma/dT$  ( $\text{mN m}^{-1} \text{ } ^{\circ}\text{C}^{-1}$ ) is the temperature coefficient of the

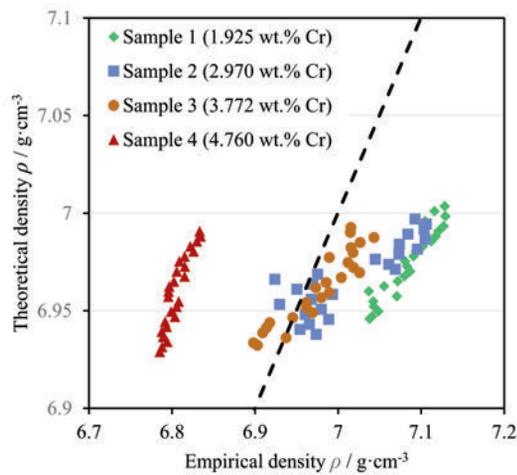


Figure 3. Comparison of experimentally obtained density values with calculated data

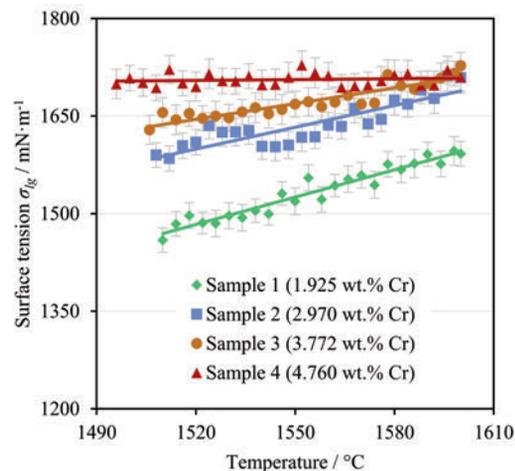


Figure 4. Surface tension values measured by means of sessile drop method at temperatures between liquidus temperature and 1600  $^{\circ}\text{C}$  (error bars denote standard error)

surface tension. The values of the calculated parameters for individual samples are summarized in Table 3.

The influence of chromium content on surface tension was assessed by the Kruskal-Wallis test [24], which revealed that the effect was statistically significant ( $p\text{-value} \ll 0.001$ ). The differences in surface tension were statistically significant at all observed chromium levels. Figure 4 shows that the surface tension of the examined samples increased with the higher content of chromium. This is probably due to its strong affinity for oxygen, which results in reduced oxygen activity and increased surface tension. Comparable results were obtained in article [9].

Another significant quantity obtained by a sessile drop method is the wetting angle, i.e. the contact angle between the Fe-alloy and the alumina substrate. The temperature dependencies of wetting angles are shown in Figure 6. The rising temperature caused a slight increase in wetting angle values. Besides, they also increased with a higher chromium content in the studied samples (Figure 7).

The interfacial tension ( $\sigma_{sl}$ ) between the molten Fe-alloy and the solid alumina substrate was calculated using Young's equation (3). This equation involves experimentally determined surface tension values ( $\sigma_{lg}$ ) of the examined steel samples, the wetting

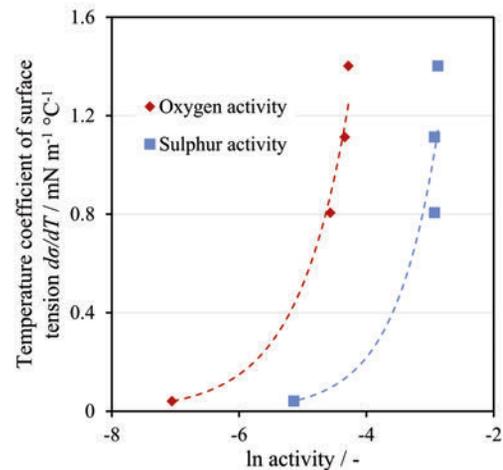


Figure 5. Variation of the temperature coefficient of surface tension with the activity of oxygen and sulfur

Table 3. Calculated parameters of linear dependence of surface tension on temperature for alloy samples 1-4

Sample	$T_{ref} / ^{\circ}\text{C}$	$\sigma_{ref} / \text{mN m}^{-1}$	$d\sigma/dT / \text{mN m}^{-1} \text{ } ^{\circ}\text{C}^{-1}$	$\Delta T / ^{\circ}\text{C}$
1	1510	1 459	$14.02 \times 10^{-1}$	1510-1600
2	1508	1 590	$11.13 \times 10^{-1}$	1508-1600
3	1506	1 629	$8.06 \times 10^{-1}$	1506-1600
4	1496	1 699	$0.41 \times 10^{-1}$	1496-1600



angles ( $\theta$ ) between the steel melt and alumina, and the surface tension of the alumina substrate ( $\sigma_{sg}$ ):

$$\sigma_{sl} = \sigma_{sg} - \sigma_{lg} \cdot \cos\theta \quad (3)$$

where  $\sigma_{sl}$  is interfacial tension ( $\text{mN m}^{-1}$ ),  $\sigma_{sg}$  surface tension of the alumina substrate ( $\text{mN m}^{-1}$ ),  $\sigma_{lg}$  is surface tension, and  $\theta$  (deg.) denotes wetting angle.

Nogi and Ogino [25] reported that the surface tension of solid alumina was  $750 \text{ mN m}^{-1}$  at  $1600 \text{ }^\circ\text{C}$  and temperature coefficient of alumina surface tension was  $-0.1 \text{ mN m}^{-1} \text{ }^\circ\text{C}^{-1}$ . The latter quantity was acceptable in the measured temperature interval [26]. Figure 8 depicts the temperature dependence on the interfacial tension of all examined samples.

The knowledge of experimentally determined values of surface tension and wetting angles also

enabled calculation of work of adhesion ( $W_{ad} / \text{mN m}^{-1}$ ) and spreading coefficient ( $S / \text{mN m}^{-1}$ ) by Young-Dupre's Equation (4) and Harkin's relationship (Equation 5), respectively. The Harkins spreading coefficient is defined as the difference between the work of adhesion and the work of cohesion. If its value is positive, spontaneous spreading occurs; otherwise, liquid forms droplet on a solid surface [27].

$$W_{ad} = \sigma_{lg} \cdot (1 + \cos\theta) \quad (4)$$

$$S = \sigma_{sg} - \sigma_{sl} - \sigma_{lg} \quad (5)$$

Figure 9 and 10 show how work of adhesion and spreading coefficient depend on the temperature. Both quantities decline with increasing temperature and chromium content.

The surface tension of the investigated steels was

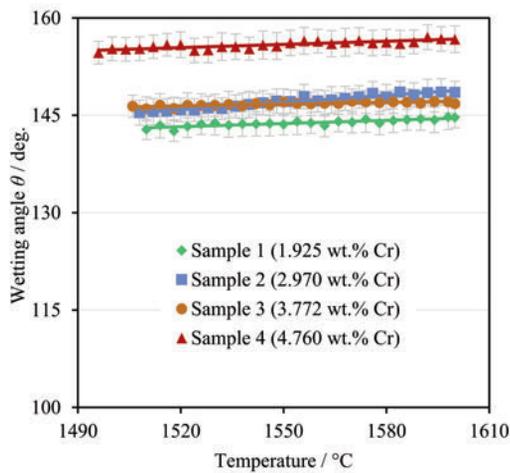


Figure 6. Dependencies of wetting angles on temperature (error bars denote standard error)

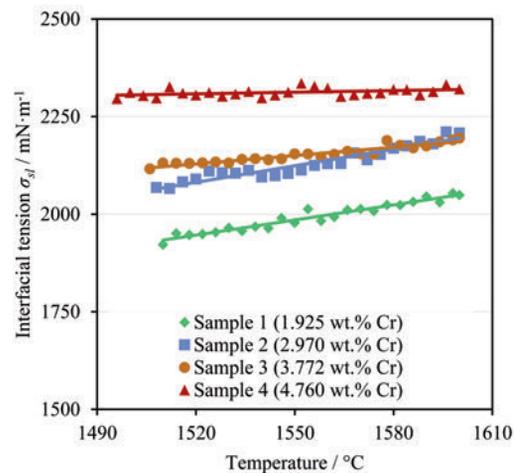


Figure 8. Interfacial tension as a function of temperature

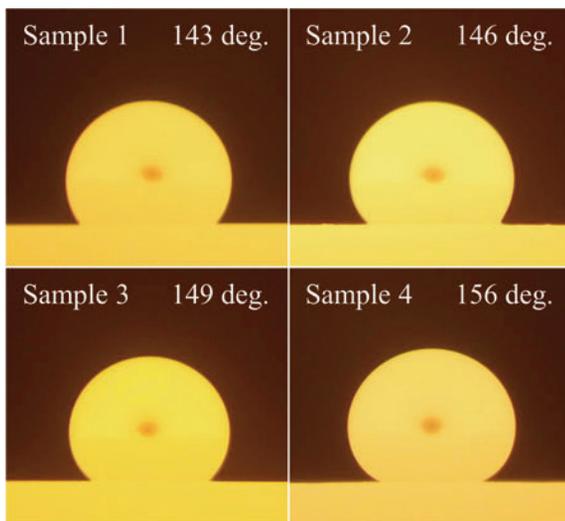


Figure 7. Images of alloy droplets taken during measurement at a temperature of  $1550 \text{ }^\circ\text{C}$

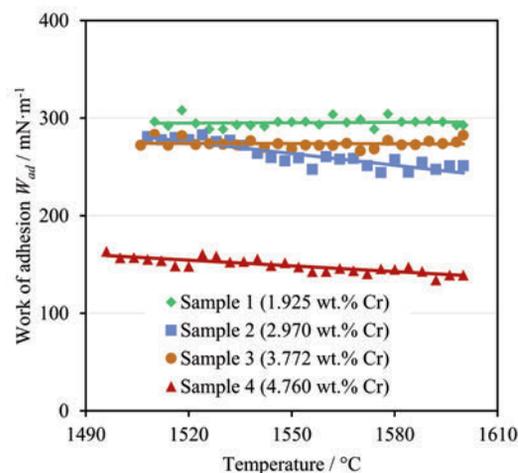


Figure 9. Dependencies of the work of adhesion on temperature

also plotted against the activity of oxygen (Figure 11) and sulfur (Figure 12) at temperatures of 1520, 1550, and 1600 °C. The figures show that surface tension decreased with increasing activity of surface-active elements. Further, at temperature 1520 °C, the surface active elements influenced surface tension on a larger scale and drop in this quantity was more pronounced, in contrast to the situation at 1600 °C. Therefore, it can be assumed that the surface of molten steel contains more surface-active elements at lower temperatures. It also confirms that as the temperature grows, these elements are desorbed into the bulk, having less effect on surface tension. Additionally, desorption caused by chromium and oxygen evaporation should be taken into consideration. As a consequence, the surface tension rises with increasing temperature.

The interaction of the steel droplet with the ceramic substrate was studied by SEM / EDS analyses for all examined samples. However, it was most significant in sample 4 which contained the highest amount of chromium (Figures 13 - 16). Based on the EDS analysis of the metal droplet, it was found that there was no dissolution of aluminum in the melt, and the free surface showed no signs of oxidation. Interaction at high temperatures resulted in the inhomogeneous surface of the ceramic plate as presented in Figures 13 and 14, where four concentric regions were identified. The area A was not affected, i.e. the reaction did not occur, however, the following elements O, Al, Ca, and Si were detected by the EDS analysis. Thus, it can be assumed that the dominant component was  $\text{Al}_2\text{O}_3$ . Also, CaO and  $\text{SiO}_2$ , the binders used in sintering, were determined, but to a lesser degree (Figures 14-15A). In addition, particles of the melt, predominantly Fe and Cr, were present in areas B and C (Figures 14-15B and 14-15C). As for the chromium, it was found that the trivalent Cr(III) could substitute Al(III) in the alumina since it had a similar atomic radius. This was also indicated by

the reddish coloring of these areas as chromium had two strong adsorption bands in the visible spectrum [28]. There was also an increased calcium content in the C area. The increase in calcium was most pronounced in area D (Figures 14-15D), where the metal drop was located, and the interaction altered the substrate most substantially. As the calcium-rich particles had a hexagonal crystal structure, calcium was presumably involved in the formation of high-temperature mineral hibonite ( $\text{CaAl}_{12}\text{O}_{19}$ ). Above mentioned conclusions are also confirmed by Figure 16. Towards the metal droplet, there was a rise of calcium content and decline in aluminum content, which correspond to the occurrence of hibonite, especially in the area under the metal droplet. The silicon content was more or less the same in all regions. The increased presence of chromium in the B and C regions correlated with the occurrence of the metallic melt.

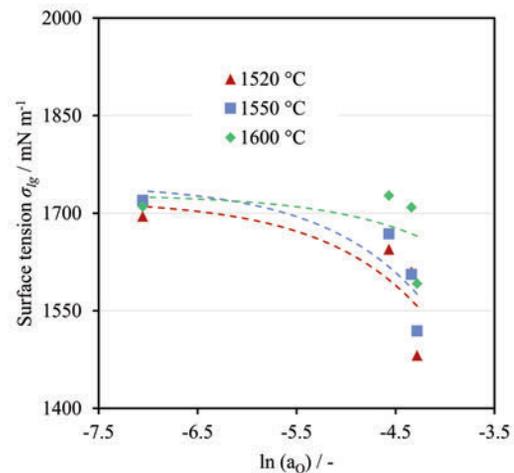


Figure 11. Surface tension as a function of the logarithm of oxygen activity at selected temperatures

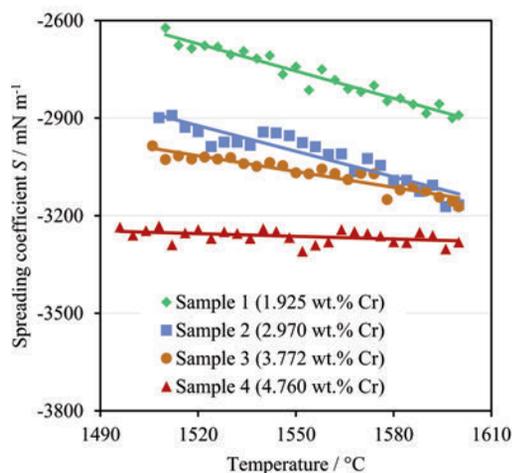


Figure 10. Temperature dependencies of the spreading coefficient

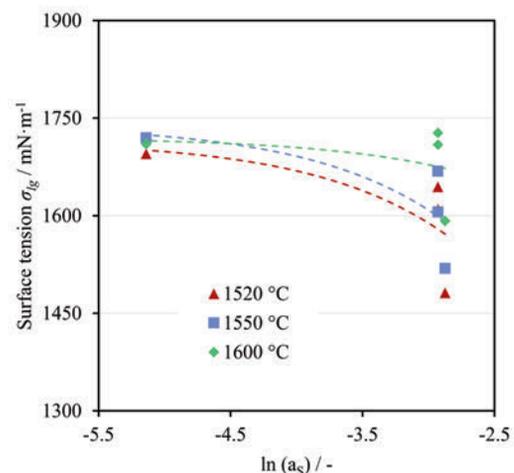


Figure 12. Surface tension as a function of the logarithm of sulfur activity at selected temperatures



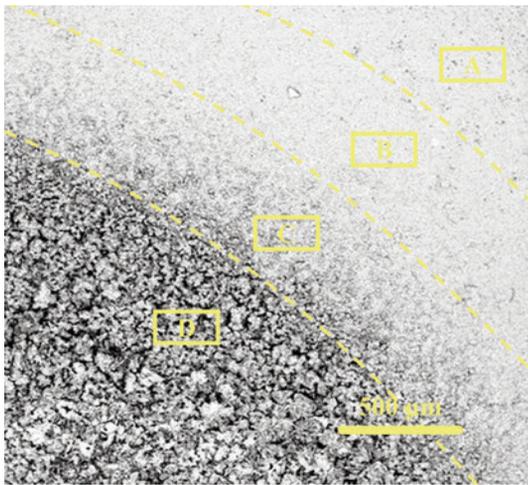


Figure 13. Macroscopic SEM image of the substrate surface after the high-temperature test (sample 4)

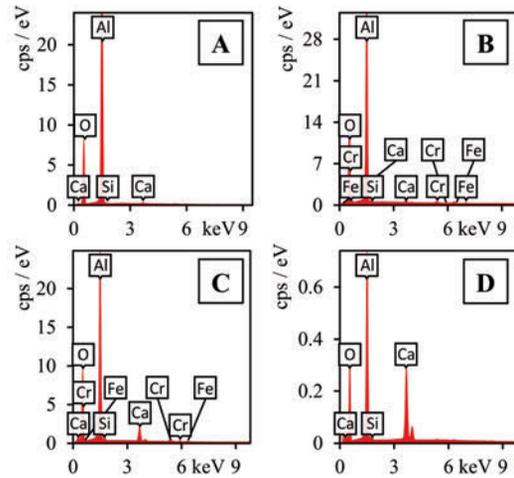


Figure 15. EDS spectra of the areas (A, B, C and D) featured in figure 13

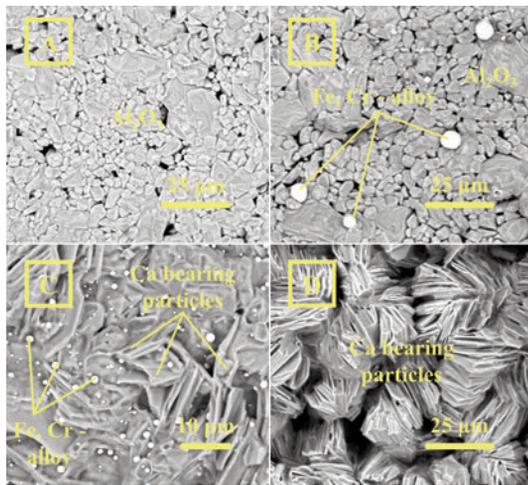


Figure 14. Magnified SEM images of zones A (unreacted area), B (Fe, Cr-rich area), C (Ca-rich area), D (reacted area)

#### 4. Conclusions

The influence of temperature and chromium content on the surface and interface properties of four model alloys was assessed in this paper. These dependencies were studied in a concentration range of 1.93 – 4.80 wt.%. The following conclusions can be drawn from this experimental research:

The linear decrease in the density of the examined alloy samples with increasing temperature was observed. Density also declined with an increase of chromium content in the samples. The comparison of experimentally determined density values with theoretically calculated by Thermo-Calc software showed a small relative error not exceeding 3.5 %.

The surface tension of the samples in contact with

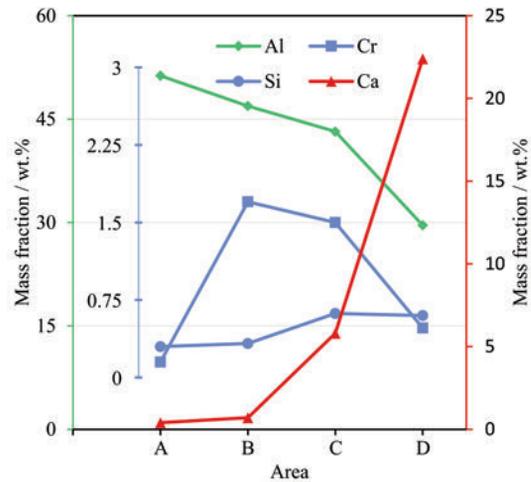


Figure 16. Changes in the mass fraction of particular elements assessed by the EDS analysis

Al<sub>2</sub>O<sub>3</sub> substrate showed a linear increase as a function of temperature. Positive temperature coefficient of surface tension can be explained by a higher content of surface-active elements (oxygen and sulfur). Besides, the chromium content increased the surface tension values since this element had a relatively strong affinity for oxygen, with the result that oxygen activity in the melt was reduced and consequently surface tension increased.

The contact angle (wetting angle) between the alloy samples and the alumina substrate depend on the temperature and chromium content, i.e. it increased with their rise.

Further, as the temperature and chromium content increased, the interfacial tension between the steel melt and the alumina substrate increased as well, unlike the work of adhesion and the spreading coefficient.

The surface tension depended on the activity of



the surface-active elements, decreasing with the increase of oxygen and sulfur activity.

Upon high temperatures, the interaction between alumina substrate and alloy samples resulted in the formation of high-temperature mineral hibonite in the area under the droplet. The calcium content raised toward the metal droplet, reaching the highest values in the area below it. In addition, a higher amount of iron and chromium was detected in the area close to the metal droplet by the EDS analysis.

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## OSOBI NE Fe-C-O-Cr LEGURA NA POVRŠINI I NA GRANIČNOJ POVRŠINI U KONTAKTU SA ALUMINIJUM

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### Apstrakt

U ovom radu je ispitivana zavisnost gustine i površinskog napona od temperature i koncentracije kod legure Fe-C-O-Cr u tečnom stanju (1.93 - 4.76 wt.% Cr) u peći za ispitivanje na visokim temperaturama putem sessile drop metode prilikom zagrevanja od likvidus temperature do 1600 °C. Osobine na graničnoj površini ( napon na graničnoj površini, ugao kvašenja, adhezija i koeficijent širenja) legura sa sadržajem aluminijuma u tečnom stanju su utvrđene u zavisnosti od temperature. Uticaj temperature i sadržaja hroma na površinske osobine, kao i svojstva na graničnoj površini je dokazan kod svih ispitivanih legura. Na osnovu činjenice da je sadržaj aktivnih elemenata kao što su kiseonik (do 195 ppm) i sumpor (do 545 ppm) na površini bio viši, procenjen je i njihov uticaj na površinski napon kod uzoraka legura. Posebna pažnja je posvećena zavisnosti površinskog napona i koeficijenta temperature od aktivnosti kiseonika i sumpora.

**Ključne reči:** Fe-C-O-Cr legura; Površinski napon i napon na graničnoj površini; Gustina; Ugao kvašenja; Efekat hroma; Sessile drop metoda.

