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Short communication

Effects of V_2O_5 addition on the corrosion resistance of andalusite-based low-cement castables with molten Al-alloy

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Abstract

Interfacial reactions between Al-alloy and andalusite low-cement castables (LCC) containing 5 wt% V_2O_5 were analyzed at 850 °C and 1160 °C using the Alcoa cup test. Interfacial reaction products and phases formed during heat treatment of the refractory samples were characterized using scanning electron microscopy (SEM) coupled with energy dispersive spectrometry (EDS) and X-ray diffraction (XRD) analysis. V_2O_5 addition resulted in the formation of glassy phases, which significantly improved the corrosion resistance. These phases were preferentially corroded by the alloy, due to their glassy nature. However, vanadium formed from reduction, formed intermetallic alloys (V–Al–Si–Mg), which formed an interfacial physical barrier to further alloy penetration.

Keywords: Interfacial reaction; Corrosion resistance; V_2O_5; Andalusite LCC refractories; Glassy phase

1. Introduction

The presence of silica in refractories used for casting aluminium alloys is often unavoidable owing to its positive effects on improving the mechanical properties; even though it is well known to be responsible for corrosive reactions with molten aluminium alloys.[1] Non-wetting agents (NWA; $BaSO_4$, CaF_2,[2] wollastonite,[3–5] AlF_3, aluminum borate,[6] aluminum titanate, cryolite,[7,8] barium celsian, [9–11] ceria, [12] BN, B_4C and B_2O_3,[13]) have been commonly added to overcome the negative effect of silica, and thus improve the corrosion resistance of refractories against molten metal.[2] The phases formed from NWA reaction with silica are thermodynamically more stable than free silica, thus improving the corrosion resistance.[14] This paper reports the effect of V_2O_5 addition on the corrosion resistance of andalusite LCC refractory with molten Al alloy. V_2O_5 has been considered for this study due to its impact as a pore blocker, and due to its effect on enhancing mullite formation in aluminosilicate refractories by removal of free silica from the refractory matrix.[15–18] Also, this research may provide a new method in utilizing the vast resources of fly-ash waste (containing considerable amount of vanadium as well as alumina and silica) from power plants in the production of low-cost aluminosilicate castable refractory for usage in aluminum melting and holding furnaces.

2. Material and methods

An andalusite LCC refractory was chosen as the base material, and its properties are listed in Tables 1 and 2. The testing of refractory corrosion resistance (extent of metal penetration) was carried out at using the Alcoa cup test at 850 °C for 150 h and 1160 °C for 75 h. The method of preparing the test samples, as well the procedure for the Alcoa cup test are listed in our previous publications.[19,20] The percentage of additive (V_2O_5) in andalusite LCC was fixed to 5 wt% since this is close to the levels used conventionally in industries where the costs of raw materials are calculated/unit weight. All raw materials for the tests were provided by Amol Refractory Industries Company, Iran, and were of industrial purity.

3. Results and discussion

Fig. 1 shows the images of the sectioned samples which were tested at different temperatures. These figures show that

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Table 1
Chemical composition of base refractory castable (no additive).

Al_2O_3	SiO_2	CaO	MgO	K_2O	Fe_2O_3	TiO_2	Na_2O	Others
61.46	31.12	2.77	0.34	1.23	0.93	0.2	0.38	1.5

Table 2
Mineralogical composition of refractory sample containing 5 wt% V_2O_5 as additive.

Material	Andalusite				Additive	R.A	M.S and cement		STTP
Size range (mm)	(3–5)	(1–3)	(0–1)	0.16	–	<0.003	0.00015	<0.003	–
Weight%	15	25	30	5	5	10	10		0.1

And: andalusite, R.A: reactive alumina, M.S: microsilica, Cement: calcium cement, STPP: tree poly phosphate sodium.

base refractory (without additives) has undergone extensive corrosion by molten aluminum (average penetration amount; at least 10 mm). However samples containing V_2O_5 showed significant improvement in corrosion resistance to Al-alloy penetration (average penetration amount; maximum 1 mm). Fig. 2 shows the XRD analysis of the refractory materials used. The samples were pre-fired at temperatures varying from 600 to 1100 °C for 5 h. The base refractory (Fig. 2a) appears to contain andalusite as the major phase along with kyanite, corundum, anorthite and cristobalite as minors. Andalusite and kyanites are polymorphs which tend to coexist naturally[21]; anorthite is formed from reactions between cement phases and

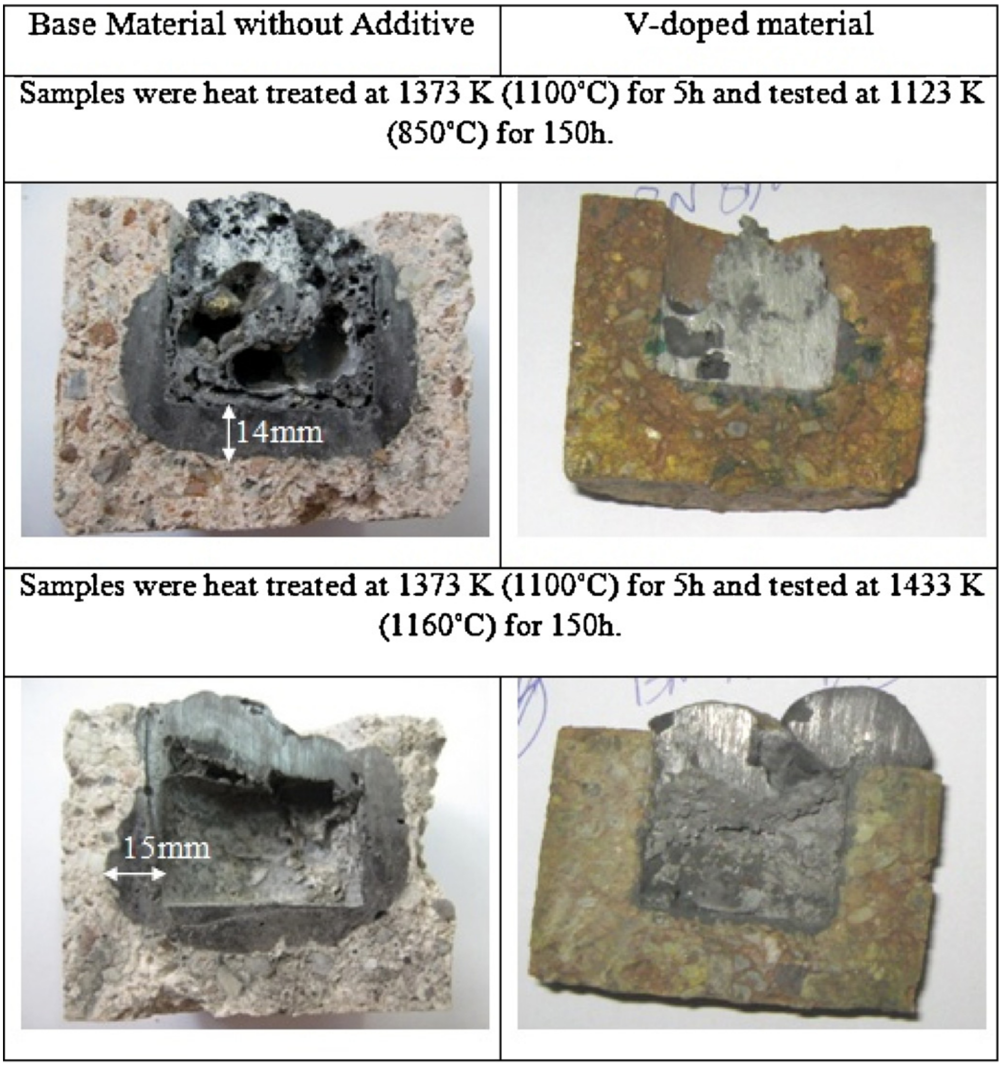


Fig. 1. Images of the samples after corrosion resistance tests.

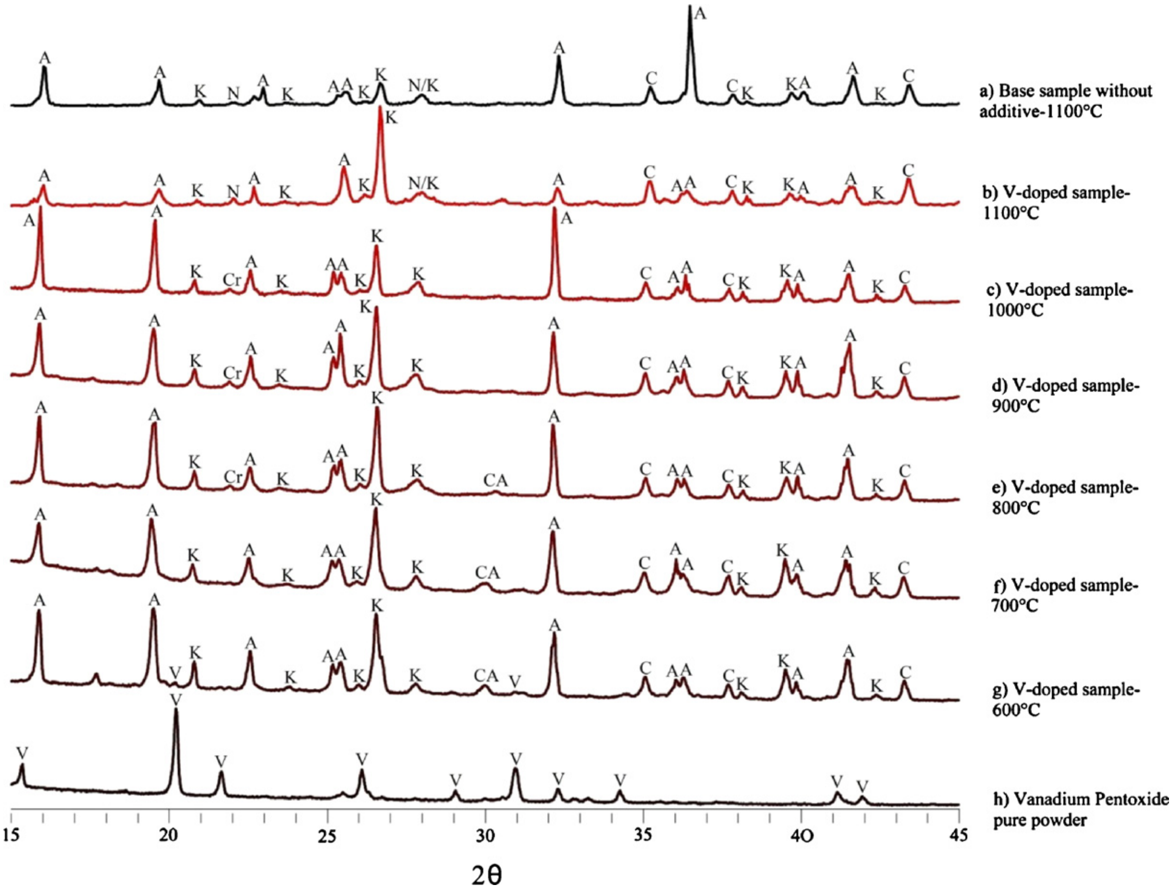


Fig. 2. XRD patterns of the different refractory samples. A: andalusite, C: corundum, Cr: cristobalite, K: kyanite, V: V_2O_5, CA: $CaO.Al_2O_3$, N: anorthite.

microsilica,[6,20,22,23] while cristobalite forms from microsilica transformations.[1]

It is believed that the formation of anorthite can improve the corrosion resistance of refractories to molten Al-alloy reactions[20,22,23]; however the amount of anorthite was insufficient to have any significant impact on the reactions between the refractory material and molten Al-alloy. In-depth characterization of corroded samples in our previous papers[19,20] revealed the formation of alumina–aluminum composite via corrosion reactions. Fig. 3 shows the typical features of this composite formed in the interface in the interfacial layer of the sample in the absence of non-wetting additives. Furthermore, when the non-wetting agent is absent from the refractory, silica (microsilica) remains unreacted and undergoes reactions with molten Al, along with andalusite. Therefore these two factors are responsible for the observed decrease in the corrosion resistance of this sample.

Fig. 1 shows that V_2O_5 addition to andalusite LCC significantly improved the corrosion resistance to molten Al alloy reactions. However, XRD analysis (Fig. 2b) reveals the absence of any new phases due to reactions of the refractory constituents withV_2O_5 after pre-firing at 1100 °C. The crystalline phases in this sample (Fig. 2b) are similar to the one without additives (Fig. 2a). For further clarification of the reactions, samples were pre-fired to different reaction temperatures [600 °C, 700 °C, 800 °C, 900 °C and 1000 °C]. XRD analysis of these samples is shown in Fig. 2c–g, while Fig. 2h shows the XRD pattern of pure V_2O_5 powder used in this study.

V_2O_5 containing samples pre-fired at 600 °C (Fig. 2g) showed the presence of andalusite as the main phase, with kyanite and alumina as minors, along with peaks of unreacted V_2O_5, while anorthite and crystobalite were absent (since the temperatures were low). Samples pre-fired at 700 °C (Fig. 2f) show diminishingV_2O_5 peaks, which disappear with further increase in the pre-firing temperature to 800 °C (Fig. 2e). This is believed to be related to V_2O_5 melting at temperatures 700 °C.[24] XRD patterns of samples (Fig. 2c–e) heated at temperature of 800–1000 °C appear similar, with the cristobalite peak appearing in the samples. Bruhns and Fischer reported that cristobalite crystallization starts ∼800 °C in the presence of V_2O_5.[25,26]

The V_2O_5 containing refractory also contains alumina, microsilica, calcium cement and alkali oxides. Figs. 4 and 5 show the different binary phase diagrams of V_2O_5 with the other refractory constituents. V_2O_5 forms a solid solution with SiO_2 below 670 °C and a liquid phase at higher temperatures (Fig. 4a[27]). V_2O_5 reacts with alumina[28–30] to form alumina vanadate ($AlVO_4$) (Fig. 4b). However, this is dependent on the source of alumina (nitrate, alkoxide),[31] heating and annealing times[32] and also production route. In our study, $AlVO_4$ formation was not seen probably since corundum has a low

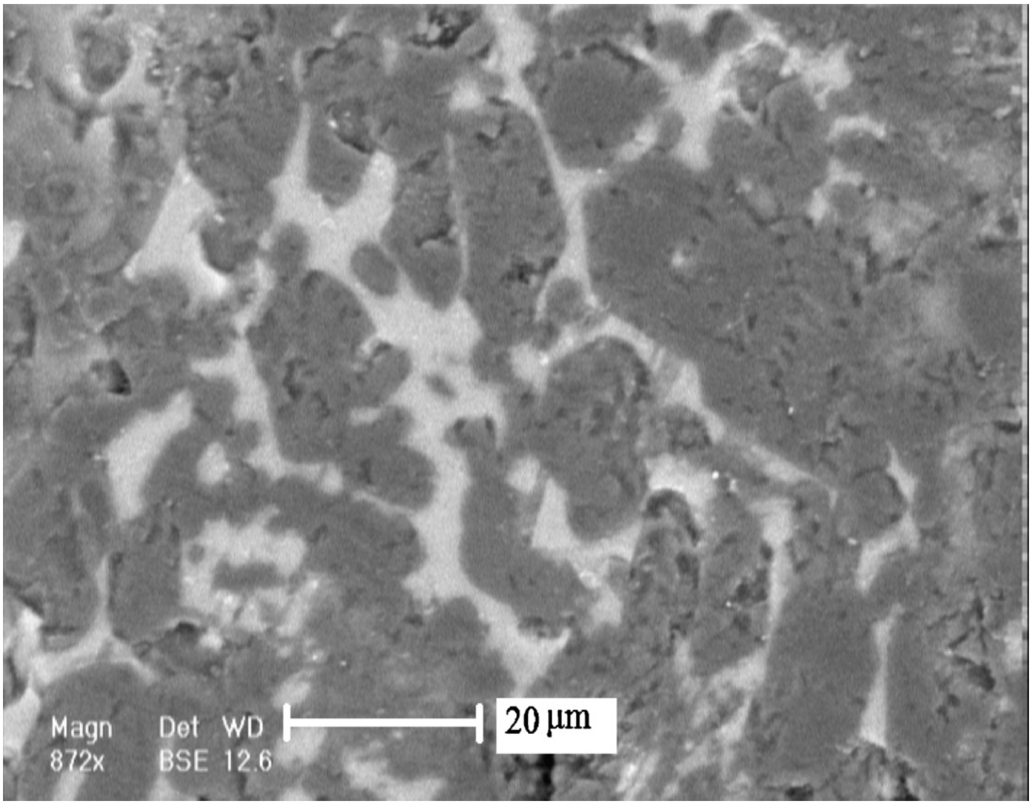
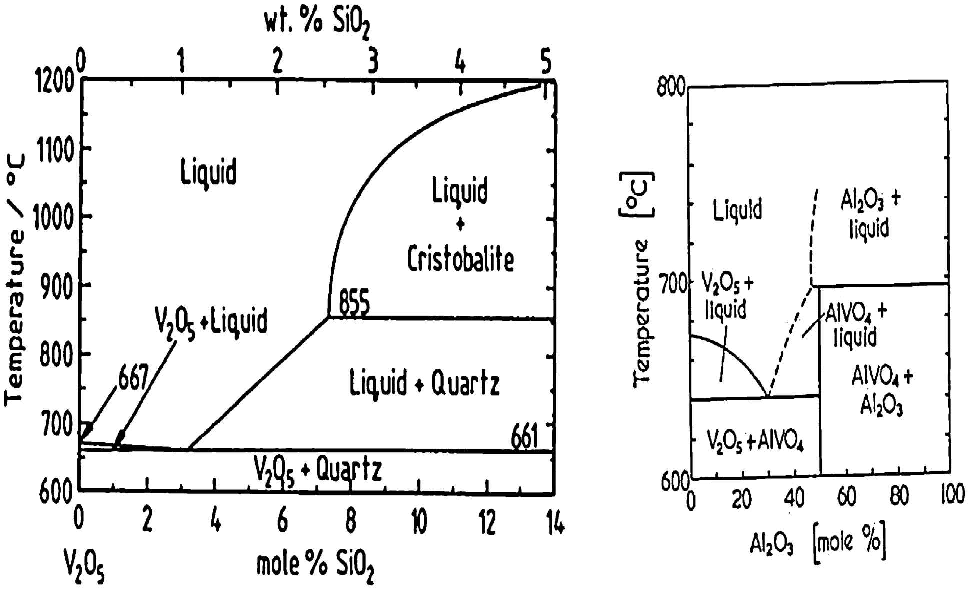


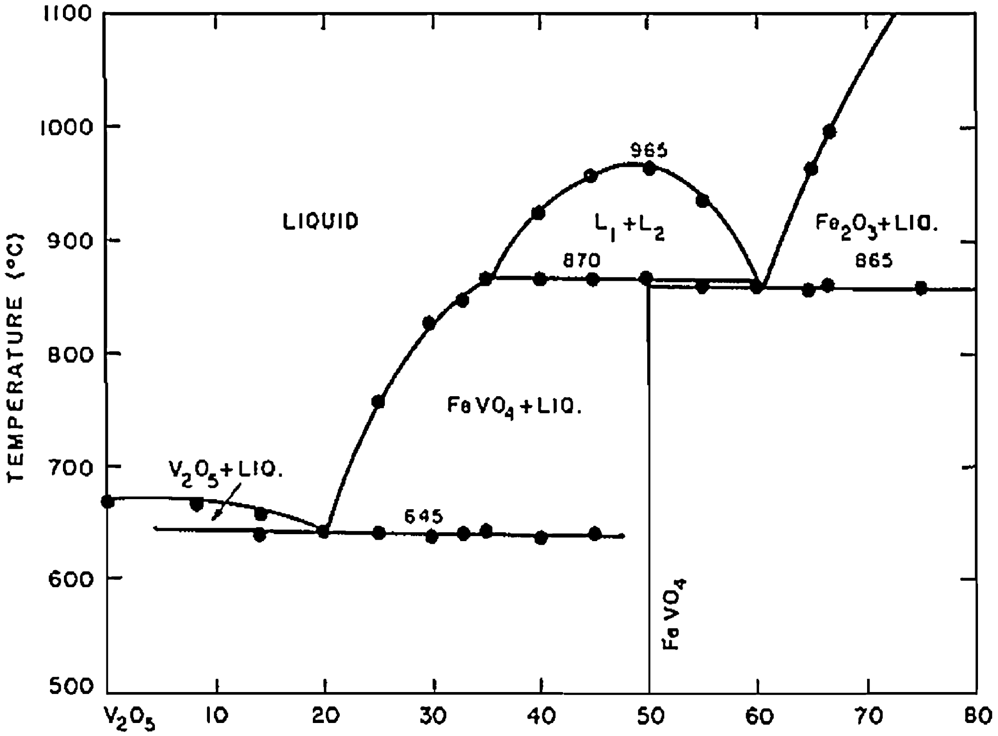
Fig. 3. BSE image of corroded area of sample without any additive (base refractory), showing the typical alumina–aluminum composite formation in the interfacial layer. Lighter colored regions are molten Al alloy containing Si and Fe, while darker regions are fresh corundum formed as a result of silica and andalusite reduction.



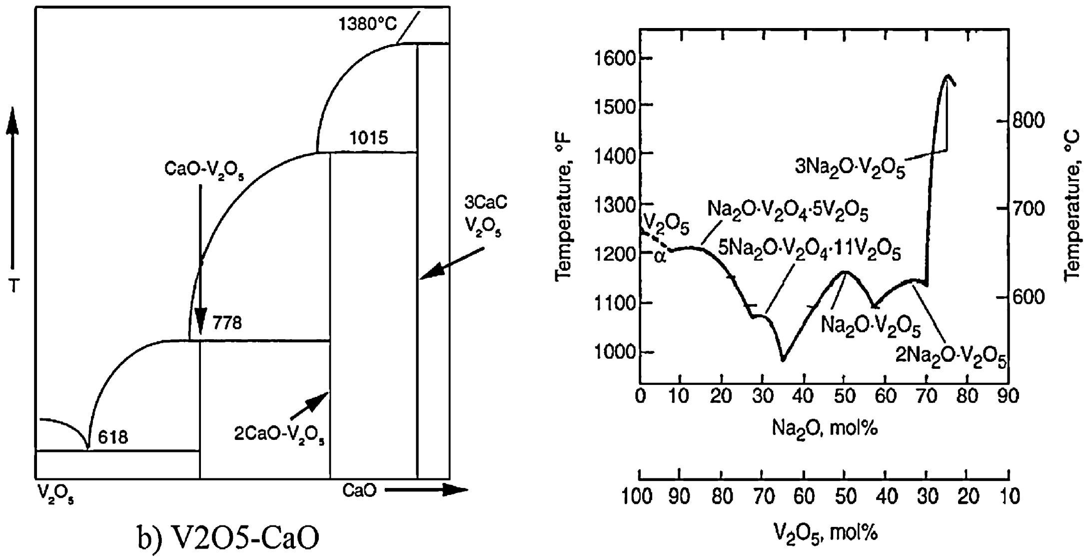
a) V2O5-SiO2

b) V2O5-Al2O3

Fig. 4. Binary phases diagram of V_2O_5–SiO_2[27] and V_2O_5–Al_2O_3..[30]



a) V2O5-Fe2O3



b) V2O5-CaO

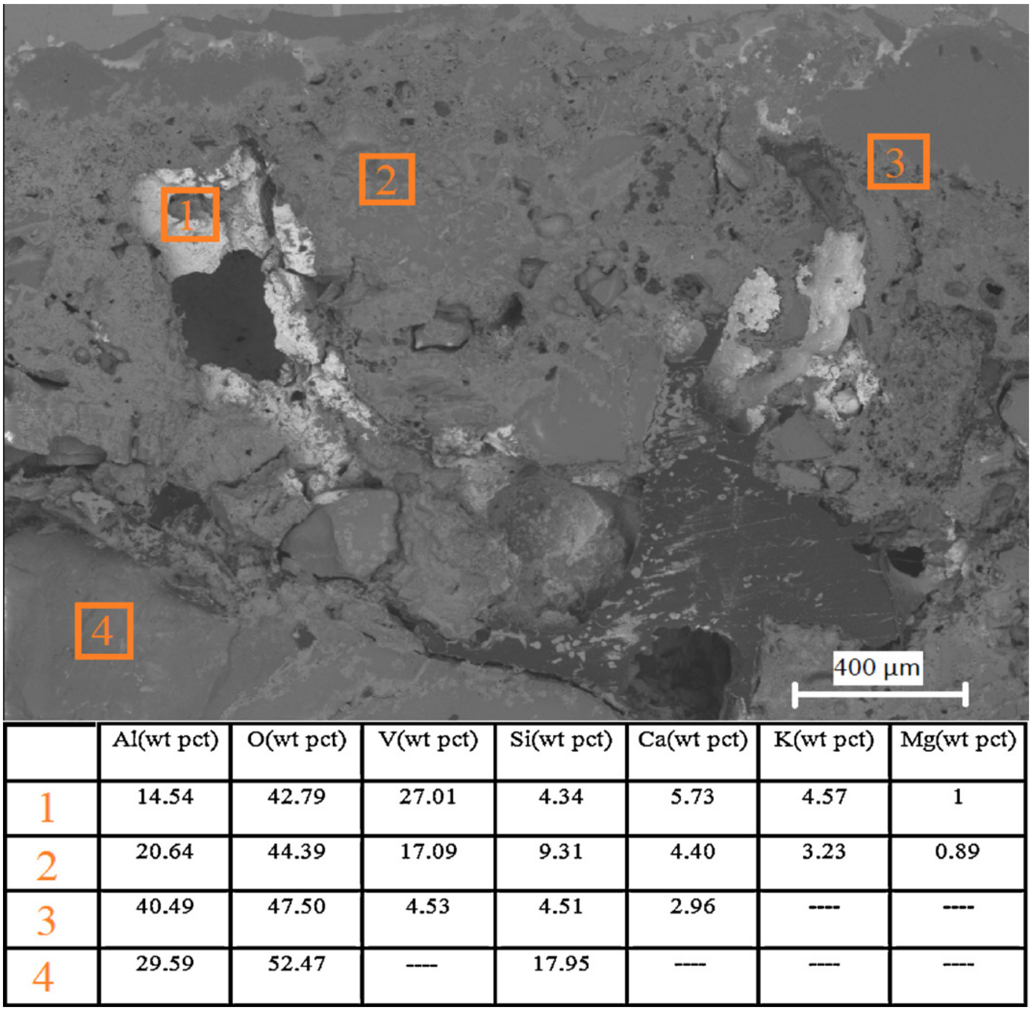
c) V2O5-Na2O

Fig. 5. Binary phases diagram of V_2O_5–CaO,[33] V_2O_5–Fe_2O_3 and V_2O_5–Na_2O.[34]

reactivity[30] and the pre-firing times were insufficient for the kinetics of aluminium vanadate formation.[32] Phase diagrams of V_2O_5–CaO,[33] V_2O_5–Na_2O and V_2O_5–Fe_2O_3[34] (Fig. 5a–c) show that the phases formed are liquid at our pre-firing and testing temperatures, and thus would not be seen in the XRD patterns.

Fig. 6 shows the EDS analysis of the microstructural region of sample containing V_2O_5. From the EDS results, white colored regions (similar to point 1) can be identified to be vanadium-rich regions. Point 2 in Fig. 6 has similar chemical composition to point 1, with slightly lower vanadium content. Both K_2O and MgO are present in these regions, which act as fluxes to enhance glassy phase formation by reactions with V_2O_5. Point 3 in Fig. 6 shows absence of K_2O or MgO, and thus could be glassy phases formed with calcium cement. Point 4 shows a composition similar to andalusite, without any vanadium presence.

Rigby and Hutton[35] and McLaren and Richardson[36] investigated interactions between V_2O_5 and aluminosilicate



	Al(wt pct)	O(wt pct)	V(wt pct)	Si(wt pct)	Ca(wt pct)	K(wt pct)	Mg(wt pct)
1	14.54	42.79	27.01	4.34	5.73	4.57	1
2	20.64	44.39	17.09	9.31	4.40	3.23	0.89
3	40.49	47.50	4.53	4.51	2.96	----	----
4	29.59	52.47	----	17.95	----	----	----

Fig. 6. EDS (point analysis) of the refractory sample containing 5 wt%V_2O_5 pre-fired at 1100 °C for 5 h.

refractories and found no reactions at different temperatures (1000–1500 °C). Others[15–18] studied the effect of V_2O_5 addition on mullite formation at different temperatures (1100–1500 °C) and concluded that a mineralizing effect existed without any chemical reactions between the constituents. Therefore, it can be concluded that V_2O_5 addition to andalusite LCC refractories forms glassy phases, while vanadium containing crystalline phases are absent (white colored regions in Fig. 6).

Fig. 7 shows the interfacial layer of the refractory sample after the corrosion test, while Fig. 8 shows the EDS analysis of this interfacial region. Spinel ($MgAl_2O_4$) layer is seen at the interface of molten metal and the refractory due to silica reduction by Mg (from the alloy) and subsequent reaction of MgO with Al_2O_3.[19,37] Spinel formation tends to cause an initial reduction of the oxides present in the refractory, which tends to make the refractory more prone to aluminium attack.[22] However, spinel formation at the interface can also retard further penetration of metal into the refractory channels.[38] From Figs. 7 and 8, it can be seen that a region with high V-content exists close to the spinel layer. EDS analysis of this interface (Figs. 7 and 8) show that this region mainly contains V, Mg and Si. Silicon and vanadium have formed from reduction of silica and vanadium oxide by molten aluminum. Also, from the elemental analysis at the interface, thin layers (50–70 µm) where segregation of V–Al–Si has occurred are seen (Fig. 8). EDS analysis show that these layers are composed of intermetallics with very low oxygen contents.

Phase diagram of V with metallic elements involved in this study (V–Al,[39] V–Si,[40] V–Mg[41] and V–Al–Si[42]) show that with increasing vanadium content in the alloys, the melting point of the alloy increases, due to high melting point of pure vanadium (1890 °C). Phase diagrams of Al–V[39] and Si–V[40] show that vanadium has low solubility in molten silicon and aluminum. Once V-concentration exceeds 15 wt% in molten aluminum, intermetallics ($Al_{21}V_2$) are formed.[39] Also, from the silicon-vanadium binary system, it is seen that these two metallic elements form a eutectic at 5.3 at% vanadium.[39] The Mg–V binary system shows that there is no restriction for the solubility of vanadium in Mg.[40] Therefore, the interfacial layers observed could be either solid solutions or intermetallics of alloys containing vanadium, or a combination of both. However,

Table 3
Gibbs free energy of the reactions involved in refractory corrosion.

Reactions	No.	Standard Gibbs free energy (ΔG°)
$2Al_{(L)} + 3V_2O_{5(L)} \rightarrow 6VO_{2(S)} + Al_2O_{3(S)}$	(1)	$(\Delta G^\circ{}_{1123\,K(850\,^\circ C)} = -1188.611\,kJ)$
$2Al_{(L)} + 1.5VO_{2(S)} \rightarrow Al_2O_{3(S)} + 1.5V_{(S)}$	(2)	$(\Delta G^\circ{}_{1123\,K(850\,^\circ C)} = -537.955\,kJ)$
$1.5SiO_{2(S)} + 2Al_{(L)} \rightarrow 1.5Si_{(L)} + Al_2O_{3(S)}$	(3)	$(\Delta G^\circ{}_{1123\,K(850\,^\circ C)} = -256.452\,kJ)$
$2Al_{(L)} + 3K_2O_{(L)} \rightarrow Al_2O_{3(S)} + 6\,K_{(L)}$	(4)	$(\Delta G^\circ{}_{1123\,K(850\,^\circ C)} = -687.656\,kJ)$
$Mg_{(L)} + V_2O_{5(L)} \rightarrow 2VO_{2(S)} + MgO_{(S)}$	(5)	$(\Delta G^\circ{}_{1123\,K(850\,^\circ C)} = -434.926\,kJ)$
$2Mg_{(L)} + VO_{2(S)} \rightarrow V_{(S)} + 2MgO_{(S)}$	(6)	$(\Delta G^\circ{}_{1123\,K(850\,^\circ C)} = -436.082\,kJ)$
$SiO_{2(S)} + 2Mg_{(L)} \rightarrow Si_{(L)} + 2MgO_{(S)}$	(7)	$(\Delta G^\circ{}_{1123\,K(850\,^\circ C)} = -248.413\,kJ)$
$K_2O_{(L)} + Mg_{(L)} \rightarrow 2\,K_{(L)} + MgO_{(S)}$	(8)	$(\Delta G^\circ{}_{1123\,K(850\,^\circ C)} = -267.941\,kJ)$

the formation of this layer acts as a physical barrier to significantly prevent refractory corrosion since these intermetallics tend to be high melting and dense at the test temperatures (850 °C and 1160 °C).

From Fig. 7, it can be seen that large metallic precipitates are present at the interface. EDS analysis shows that these precipitates contain Al (61.38 wt%), Si (9.62 wt%), Fe (22.55 wt%) and V (6.43 wt%). However no iron or vanadium was observed within the molten metal, which contained only aluminium and silicon (from reduction).

On the basis of presented results, V_2O_5 is observed to form a glassy phase with the refractory constituents at temperatures of 600–1100 °C. When this refractory reacts with molten Al-alloy, the glassy phases are the first to react with the corrosive elements (Al/Mg). V_2O_5 and other possible constituents of the glassy phase are reduced by molten Al-alloy, and the respective equations and their corresponding Gibbs free energies are listed in Table 3. V_2O_5 is the first component of the refractory that is thermodynamically reduced by corrosive elements (Al/Mg). After V_2O_5 reduction, vanadium segregates in the interfacial layer, and reacts with Si (formed from SiO_2 reduction), and the alloy constituents to form intermetallics, which have been confirmed to form at the interface from EDS analysis. These compounds are high-melting and tend to saturate the molten metal, thus forming a barrier to further corrosion.

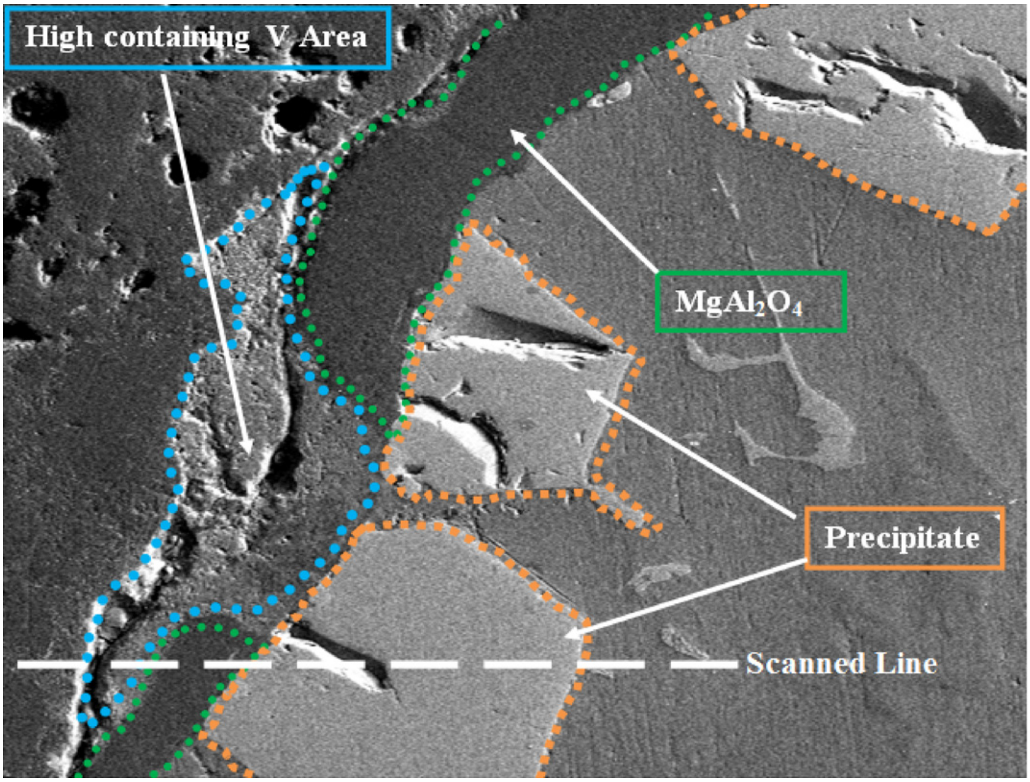


Fig. 7. The interfacial layer of V_2O_5 containing refractory samples (pre-fired at 1100 °C for 5 h and tested at 850 °C for 150 h).

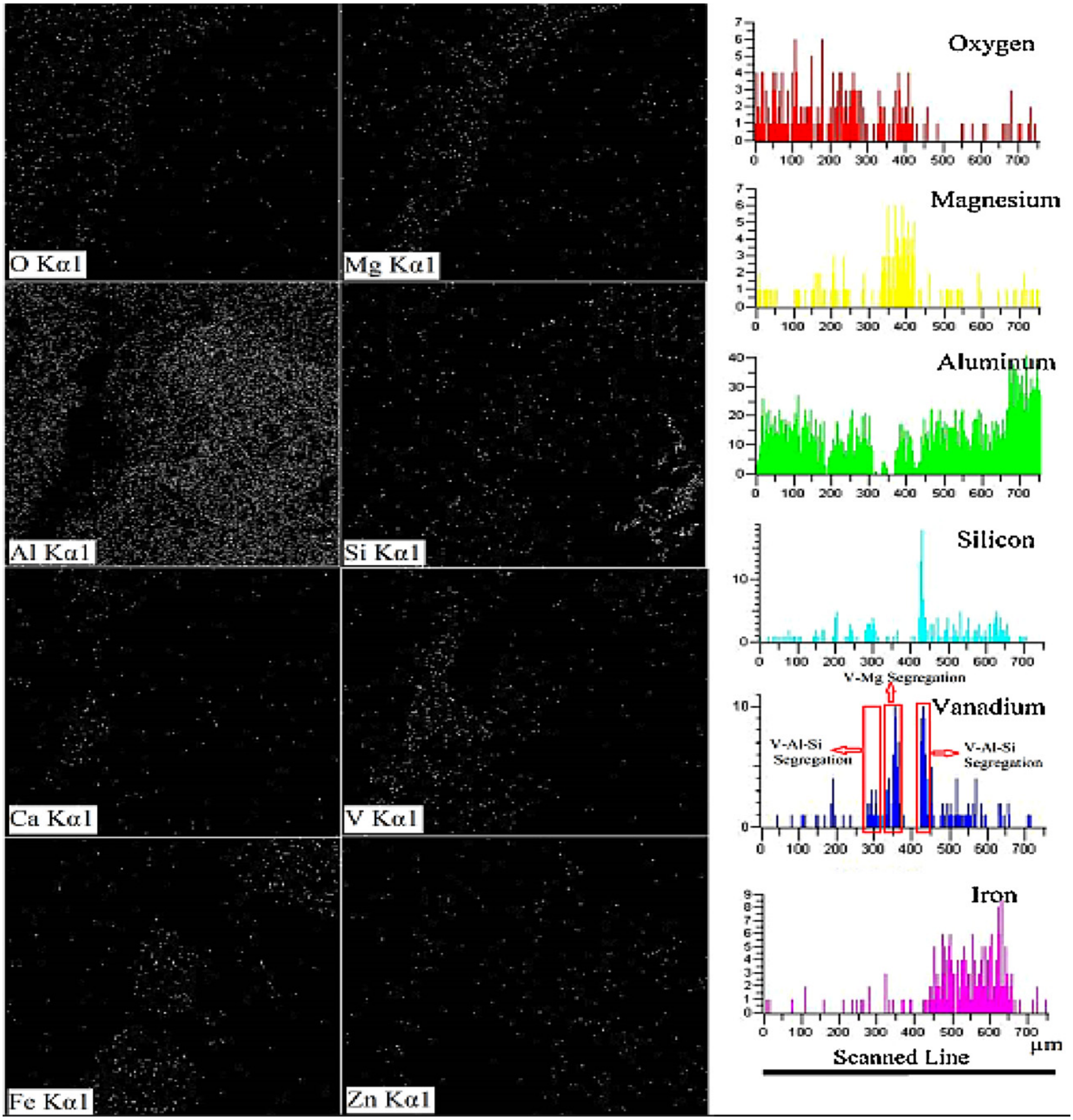


Fig. 8. Elemental mapping and line analysis of the interfacial region shown in Fig. 6 (elemental line scan for vanadium shows formation of segregated intermetallic regions).

4. Conclusion

This study investigated the corrosion resistance of andalusite LCCs containing vanadium pentoxide as additive material. The major findings are as follows:

- Addition of vanadium pentoxide (V_2O_5) has been shown to significantly improve the corrosion resistance of andalusite LCC refractories.
- V_2O_5 addition resulted in the formation of glassy phases in different pre-heating temperature, which significantly improved the corrosion resistance. These phases were preferentially corroded by the alloy, due to their glassy nature.
- Formation of intermetallic compounds (formed from V, Si, Al and Mg) at interfacial layer contribute to lowering the corrosion of sample containing V_2O_5, mainly due to the high stability and melting point of these compounds as physical barrier against to further molten alloy penetration.

Acknowledgments

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