

CHARACTERIZATION OF NON-METALLIC INCLUSIONS IN HOT ROLLED CALCIUM TREATED STEEL

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ABSTRACT

A study of the relationship between inclusions' shape and composition in hot-rolled calcium treated industrial steel was undertaken in this work. Scanning electron microscope equipped with an energy dispersive X-ray (EDX) analyzer was used to analyze the elemental composition of the inclusions. The analyzed elemental compositions were converted into their respective thermodynamically most stable oxides and sulphides, and appropriate phase diagrams were used to determine their formulae. Results obtained reveal that complex oxysulphides and $\text{CaO}\cdot\text{Al}_2\text{O}_3$ compound of very small sizes remain undeformed. $12\text{CaO}\cdot 7\text{Al}_2\text{O}_3$ compound deformed and fractured into strings. $12\text{CaO}\cdot 7\text{Al}_2\text{O}_3$ containing small amount of $\text{CaO}\cdot\text{Al}_2\text{O}_3$ deformed plastically. While inclusions that deform into short plastic threads and brittle strings mixed together are made up of small amounts of $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ compound in the core of $12\text{CaO}\cdot 7\text{Al}_2\text{O}_3$. The undeformed (irregular shaped) parts of some of the inclusions were found to contain $\text{CaO}\cdot\text{Al}_2\text{O}_3$, $\text{Al}_2\text{O}_3\cdot\text{MgO}$ and $2\text{Al}_2\text{O}_3\cdot 3\text{CaO}\cdot\text{MgO}$ compounds.

KEYWORDS: Rolled strip, Inclusions, Calcium treated, Oxysulphides, Brittle strings.

INTRODUCTION

The behaviour of non-metallic inclusions in steel during working processes has continued to attract the interest of researchers because of their harmful influence on the properties of steel. Although earlier studies were centered around the amount of inclusions present in steel based on oxygen and sulphur contents, recent researchers are focusing more and more on the size, shape, distribution and chemical composition of inclusions and how some of these parameters behave during the processes of steel working and their subsequent effect on the quality of the final products. Numerous studies carried out on the behaviour of inclusions during hot working have reported that the extent of deformation of inclusions depend largely on their chemical compositions and the temperature of deformation^[1]. Very early studies on inclusions behaviour during steel working by Scheil and Schnell^[1] showed that at low temperatures, oxides of silicate inclusions were brittle but at higher temperatures, these inclusions become deformable based on the quantity of SiO_2 in the composition. The same study reported that sulphides deformed plastically independent of temperature.

The deformability of (Fe, Mn)O inclusions type have been reported as having decreased plasticity with increase in Mn content^[2,3].

The deformation behaviour of types I, II and III manganese sulphides is well established^[4]. These research findings are in agreement with that of Scheil and Schnell that manganese sulphide inclusions generally elongate without fracturing during steel working irrespective of temperature.

Other studies on silicate inclusions^[5-9] have also confirmed their brittle nature at low temperature and increased plasticity above a certain critical temperature.

Recent studies^[10-13] have dealt with the calcium aluminates type of inclusions present in steel and their deformation behaviour in some hot working processes. However, study on the effect of rolling on the deformation of calcium aluminate inclusions is still generating a lot of interest.

In this paper, inclusions shape of chemically characterized calcium aluminates found in hot worked pipeline steel are presented and explanations are given on the possible role of inclusions' composition on the nature of their deformation.

EXPERIMENTAL MATERIAL

The composition of the low alloyed pipeline steel used in the investigation is given in table 1.

Table 1 Composition of low-alloyed pipeline steel, %.

Fe	0.078-0.095C	0.21-0.276Si	1.35-1.42Mn	0.011-0.012P	0.033Nb
0.003-0.005S	0.019-0.039Cr	0.01-0.012Ni	≤0.08(N)	≤0.044V	0.004Ti
0.037-0.042Al	0.014-0.017Cu	0.003(O)			

SAMPLING TECHNIQUE

Two templates (front and back templates) were prepared from the bulk of the strip of the pipeline steel under investigation. From each template, three rectangular (20mm x 10mm) specimen blanks were machined out giving a total of six samples in conformity with the Russian standard inclusions assessment method, Gost 1778 and ASTM standard E-45.

SAMPLE PREPARATION AND ANALYSIS

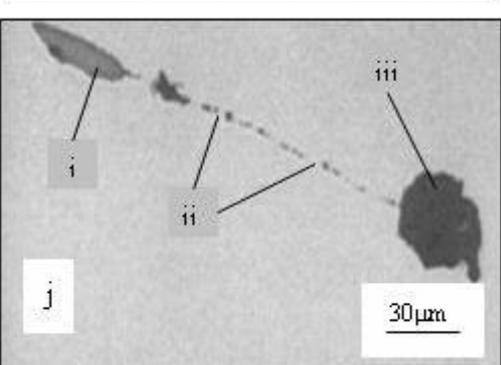
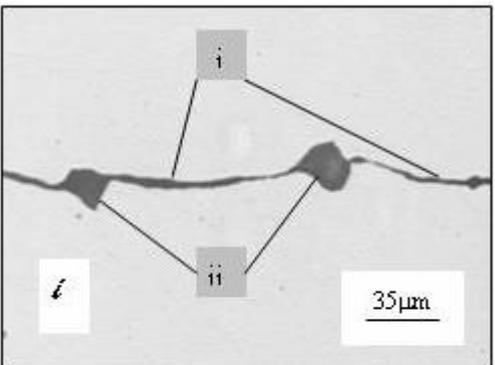
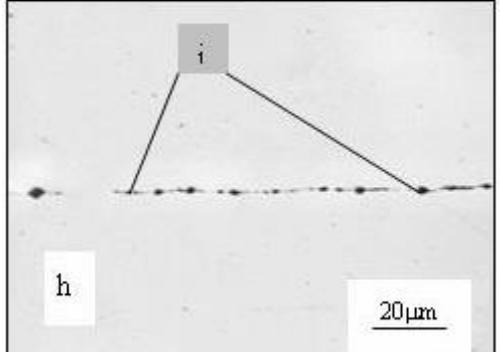
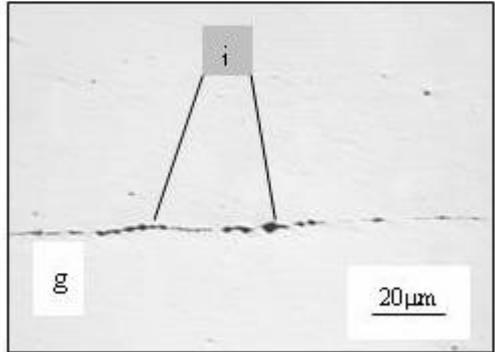
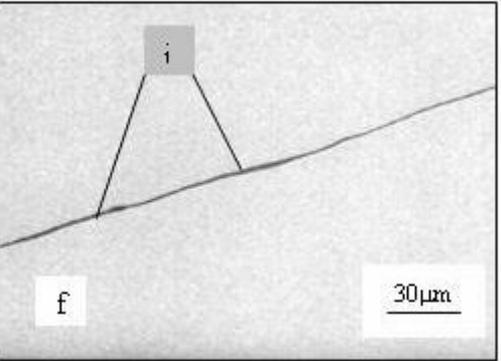
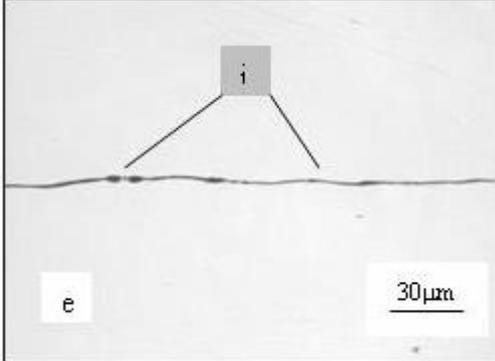
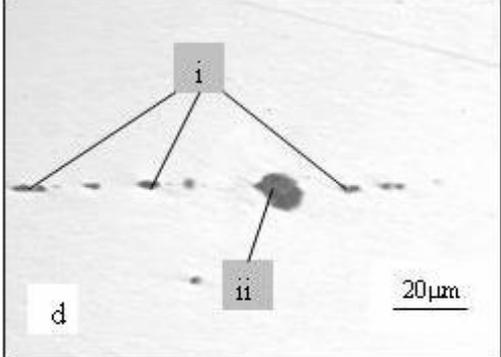
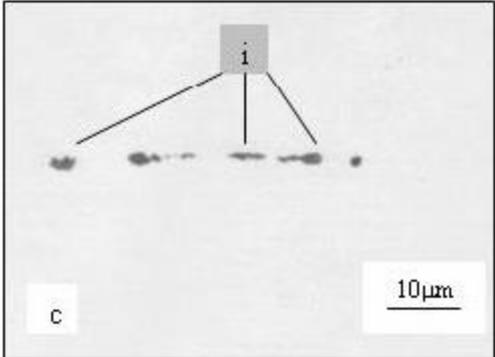
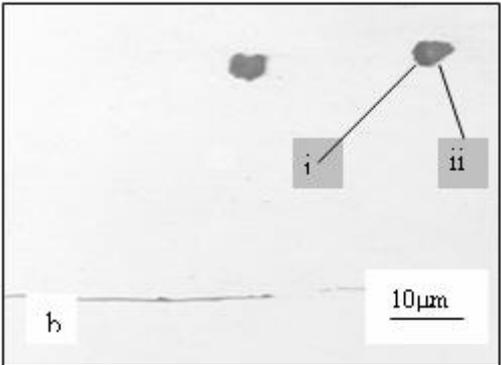
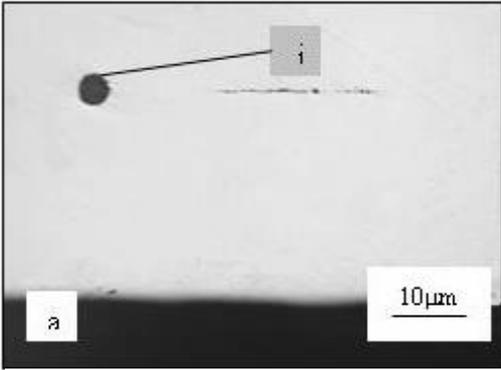
Samples for metallographic examination were ground and polished using grinding machine and different grades of emery paper sizes and alumina polishing powder suspended in distilled water. Polished samples were then examined under an optical microscope equipped with a digital camera. Based on differences in optical appearance, representative inclusions were identified and marked out for further analysis using a scanning electron microscope (SEM) – Cam Scan model equipped with an energy dispersive X-ray (EDX) analyzer (link analytical LZ-5). The analyzer was used to detect the elements, while the EDX software was used to calculate the elemental compositions of inclusions.

RESULTS AND DISCUSSION

RESULTS

Calcium aluminate inclusions observed in the steel samples based on their peculiar features as revealed by optical microscope have been classified into five main types, namely:

- Undeformed small spherical inclusions (figs.1a & 1b);
- Discontinuous strings along the rolling direction, resulting from crushed brittle inclusions (figs.1c & 1d);
- Plastically (unfractured) ductile inclusions (figs.1e & 1f);
- Mixed inclusions of plastic threads and brittle strings (figs.1g & 1h);
- Partly deformed inclusions, with the deformed parts forming ductile threads or brittle strings or both (figs. 1i – 1k).



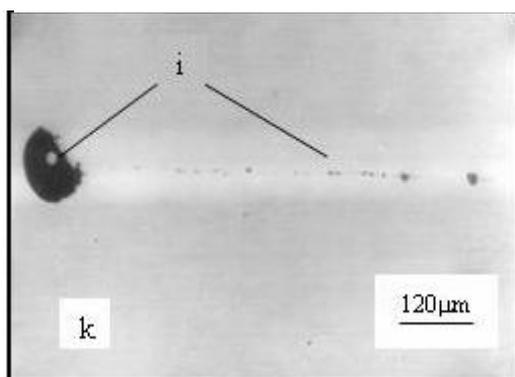


Figure 1: Micrographs of inclusions' shape in hot rolled steel.

a & b: Undeformed small spherical inclusions;

c & d: Discontinuous strings along the rolling direction, resulting from crushed brittle inclusions;

e & f: Plastically (unfractured) ductile inclusions;

g & h: Mixed inclusions of plastic threads and brittle strings;

l,j & k: Partly deformed inclusions, with the deformed parts forming ductile threads or brittle strings or both.

Table 2 Elemental compositions of inclusions' type investigated

Figure	Points analysed	Elemental Composition, %						
		Al	Ca	Mg	Mn	S	Si	Ti
1a	i	47.6	44.2	0.4	0.8	3/8	3.2	—
1b	i	28.8	30.0	2.1	18.8	19.8	0.7	—
	ii	35.0	39.0	0.7	2.8	20.0	2.2	—
1c	i	39.1	52.9	3.4	—	2.3	0.8	—
1d	i	41.0	53.5	2.4	0.1	0.1	0.3	-
	ii	35.0	39.0	0.7	2.8	20.0	2.2	-
1e	i	44.6	51.6	0.3	0.5	1.5	1.4	—
1f	i	45.0	48.7	3.9	—	0.1	1.9	—
1g	i	39.9	54.5	2.8	0.1	0.1	1.8	0.8
1h	i	44.6	51.6	0.3	0.5	1.4	1.5	—
1i	i	49.9	43.8	3.4	1.5	0.6	0.8	—
	ii	73.4	1.8	24.7	0.1	0.1	—	—
1j	i	15.6	37.4	2.8	8.9	34.8	0.5	—
	ii	42.2	53.6	2.0	0.1	0.9	1.3	—
	iii	43.7	41.8	11.7	0.3	0.1	2.2	0.8
1k	i	58.0	40.7	1.1	0.2	—	—	—

The results of the elemental analysis of inclusions using SEM are given in table 2. The elemental compositions of inclusions were converted into their respective most thermodynamically stable oxides and their stoichiometric ratios were determined. The Al₂O₃-MgO, CaO-Al₂O₃ and CaO-Al₂O₃-MgO equilibrium diagrams^[14] were used to determine their chemical formulae appended in table 3.

Table 3 Stoichiometric compositions and chemical formulae of inclusions' type investigated

Figure	Points analysed	Stoichiometric Composition, %						Chemical formula
		Al ₂ O ₃	CaO	MgO	CaS	MnS	SiO ₂	
1a	i	56	35	-	5	-	4	CaO·Al ₂ O ₃
1b	i	40	20	3	15	22	-	CaO·Al ₂ O ₃ + (Ca, Mn)S
	ii	46	19	-	28	4	3	CaO·Al ₂ O ₃ + CaO·2Al ₂ O ₃ + CaS
1c	i	53	47	-	-	-	-	12CaO·7Al ₂ O ₃
1d	i	51	49	-	-	-	-	12CaO·7Al ₂ O ₃
	ii	46	19	-	28	4	3	CaO·Al ₂ O ₃ + CaO·2Al ₂ O ₃ + CaS
1e	i	54	46	-	-	-	-	12CaO·7Al ₂ O ₃ + CaO·Al ₂ O ₃
1f	i	56	44	-	-	-	-	12CaO·7Al ₂ O ₃ + CaO·Al ₂ O ₃
1g	i	48	49	3	-	-	-	3CaO·Al ₂ O ₃ + 12CaO·7Al ₂ O ₃
1h	i	47	53	-	-	-	-	3CaO·Al ₂ O ₃ + 12CaO·7Al ₂ O ₃
1i	i	57	38	3	-	-	-	12CaO·7Al ₂ O ₃ + CaO·Al ₂ O ₃
	ii	76	-	23	-	-	-	MgO·Al ₂ O ₃
1j	i	27	-	3	58	12	-	Al ₂ O ₃ + (Ca, Mn)S
	ii	52	48	-	-	-	-	12CaO·7Al ₂ O ₃
	iii	50	35	12	-	-	3	3CaO·MgO·2Al ₂ O ₃
1k	i	66	34	-	-	-	-	CaO·Al ₂ O ₃

DISCUSSION

Results obtained revealed the presence of undeformed inclusions of very small sizes (~ 3µm) and variously deformed inclusions in the samples analysed. These inclusions were both mono and multi phased. SEM analysis results of type 1a inclusions show that they contain calcium and aluminium oxides with no noticeable gradient in the concentrations of these oxides across the volume of the inclusions. The stoichiometric composition obtained for type 1a inclusions corresponds to CaO·Al₂O₃ compound on the phase diagram of the CaO-Al₂O₃ system. Calcium aluminate of this structure is not very rich in calcium oxide and has a very high melting point of over 1600°C [7].

The undeformed inclusions of type 1b have been found to be two-phase oxysulphide particles. The dark grey phase (1b, i) contains calcium and aluminium oxides and the double sulphides of the type (Ca, Mn)S that corresponds to CaO·Al₂O₃+(Ca,Mn)S. As appended in table 3, MnS is quantitatively more in proportion than CaS. In the light grey phase (1b, ii), MnS concentration is very negligible, as the sulphide component of the phase is practically made up of CaS. This, of course, explains the lighter appearance of the phase.

Also observed were inclusions that are extensively deformed and fractured into strings (figs.1c & 1d). As previously highlighted, this characteristic behaviour of calcium aluminates is also similar to that of silicates in rolled steels. However, results from this study have restricted this behaviour to 12CaO·7Al₂O₃ type of calcium aluminates. Calcium aluminate of this structure is fairly rich in calcium oxide and has a comparatively low melting point (~1455°C) [7]; this is likely to account for its fragmentation into strings during cooling after an extensive elongation

Figures 1e and 1f show severely deformed but unfractured inclusions. A careful observation of the two figures, suggests a high ductility level of these inclusions. SEM investigation of the

inclusions indicates that they are complex calcium aluminates that combine the $12\text{CaO}\cdot 7\text{Al}_2\text{O}_3$ and $\text{CaO}\cdot \text{Al}_2\text{O}_3$ types. However, the proportion of $\text{CaO}\cdot \text{Al}_2\text{O}_3$ is very low in both instances. The likely reason for the plastic nature of the deformation of these inclusions is the presence of the $\text{CaO}\cdot \text{Al}_2\text{O}_3$ compound in the core of $12\text{CaO}\cdot 7\text{Al}_2\text{O}_3$ calcium aluminate. Although only in small amount, yet enough to enhance the ductility of $12\text{CaO}\cdot \text{Al}_2\text{O}_3$ type of calcium aluminate.

The inclusions presented in figures 1g and 1h are mixed deformed inclusions, composed of short plastic threads and brittle strings. Results of the analysis revealed that they are complex calcium aluminates, with small amounts of $3\text{CaO}\cdot \text{Al}_2\text{O}_3$ compound in the core of $12\text{CaO}\cdot 7\text{Al}_2\text{O}_3$ calcium aluminate. $3\text{CaO}\cdot \text{Al}_2\text{O}_3$ compound which is very rich in calcium oxide but has a moderately high melting point of about 1535°C [7], could be the most likely reason for the type of mixed deformation that happened after rolling.

Figure 1i combines unfractured plastic thread with undeformed, irregular shaped particles. SEM analysis showed that the composition of the plastic part (1i, i) is in agreement with that obtained for inclusions 1e and 1f, i.e. $12\text{CaO}\cdot 7\text{Al}_2\text{O}_3$ combine with small amounts of $\text{CaO}\cdot \text{Al}_2\text{O}_3$. The undeformed parts (1i, ii) have been found to contain MgO and Al_2O_3 in a proportion that approximates to the stoichiometric range of the spinel phase $\text{MgO}\cdot \text{Al}_2\text{O}_3$ on the phase diagram of the MgO- Al_2O_3 system [14]. This compound has a very high melting point of about 3135°C [7].

The inclusion presented in figure 1j has differences in optical appearance. Results obtained showed that the dark spot on the light grey phase (1j, i) contains pure aluminium oxide, while the light grey part reveals a double sulphides of the (Ca, Mn)S type, but with much higher concentration of CaS than that of MnS that resulted in the light grey appearance of the phase. An inclusion of this composition has been characterized to have melting point and hence it was only slightly deformed. The elongated parts (1j, ii) of the inclusion are tiny particles composed of single calcium aluminate whose composition is similar to that of figures 1e and 1f, i.e. $12\text{CaO}\cdot 7\text{Al}_2\text{O}_3$. The darkish undeformed, irregular shaped part (1j, iii) of the inclusion has been analysed to contain a ternary phase, made up of Al_2O_3 , CaO and MgO. The composition obtained corresponds to $2\text{Al}_2\text{O}_3\cdot 3\text{CaO}\cdot \text{MgO}$ compound on the equilibrium diagram of Al_2O_3 -CaO-MgO system [14]. The source of this inclusion is likely to be from entrapped vessel lining or slag mixture. The existence of this inclusion type has also been reported in previous researches [14, 15].

The partly crushed inclusion 1k has as its constituent a single calcium aluminate compound $\text{CaO}\cdot \text{Al}_2\text{O}_3$. Calcium aluminate compound of this composition does not deform, however, the inclusion is fairly big in size which possibly explains why a part of it crumbled during rolling.

CONCLUSION:

The investigation conducted on hot rolled industrial steel revealed the presence of undeformed high melting $\text{CaO}\cdot \text{Al}_2\text{O}_3$ type of calcium aluminates and complex oxysulphides. Also observed were inclusions deformed both plastically and in brittle forms, composed of single and complex calcium aluminates in which the low melting $12\text{CaO}\cdot 7\text{Al}_2\text{O}_3$ compound featured prominently but in different proportions. Magnesium and aluminium oxides were also found in compositions of some of the undeformed calcium aluminates.

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