

EFFECT OF RARE EARTH METALS INJECTION AND GAS STIRRING ON CLEANLINESS OF PIPELINE STEEL

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ABSTRACT

A full scale plant trial was done to find the effect of rare earth metals (RE) wire injection on deoxidation, desulphurization and inclusion of steel rolling pipe. The melt was stirred by nitrogen from the bottom of the ladle. For about 5 to 15 minutes. As a result of addition of RE at the rate of 0.2 kg/t substantial deoxidation, desulphurization and reduction of oxygen activity from about 50 to 7 ppm level took place.

Addition of 0.1 kg/t of RE resulted in difference of oxygen by 0.01278%, while the oxygen content in the bath was 0.010%. When RE addition was done over 0.03 kg/t, oxysulphides and its complex were formed depending upon RE and aluminum contents. The stirring of the melt by gas helped in reducing quantity of inclusions.

KEY WORDS: Rare earth metals, injection, deoxidation, desulphurization, inclusion, steel.

1. INTRODUCTION

The injection metallurgy begun at the end 1960's. The injection technology was very much developed in Finland since 1972. The use of the ladle injection had some achievements [1-9]. The use of rare earth metals (RE) injection to ladle was discussed in earlier works [10-18]. The RE additions for deoxidation and desulphurization of steel in the ladle were also discussed in a lot of research works [19-30]. However previous RE additions in such a steel did not appreciably reduce the total oxygen and sulphur levels. The RE additions usually resulted in oxides RE_2O_3 , oxysulphides RE_2O_2S and sulphides RE_xS_y , because these inclusions were relatively heavy. For this reason there was increase of inclusion in steel [31-35].

Hence the present work was undertaken with a view to reduce / eliminate above problems. Essentially here, the effect of RE wire injection together with gas stirring from bottom of the ladle, on the deoxidation and desulphurization of steel was done. The stirring by gas was done to effectively remove the inclusions by floatation principle [36-40]. This was a corporate research project of IFM Dobra - Czech.

The work described the effect of RE wire injection together with gas stirring on the deoxidation and desulphurization of steel rolling pipe.

2. EXPERIMENTAL

Industrial trials were performed with steel containing about 0.09-0.20%C, 0.18-0.40%Mn, 0.017-0.025%P, 0.014-0.033%S, 0.002-0.01%Al, 0.01225-0.04026%O₂(total) and 0.0018-0.01235%N₂ and this steel was produced in an 45ton-UHP electric arc furnace. The bath after the oxidizing and deslagging operations was subjected to the addition of FeMn, FeSi, Al and CaO + CaF₂. After waiting for a period necessary to allow the slag forming of about 1600°C, the steel was tapped into the ladle. After that the ladle was taken to the injection station. The temperature and oxygen activity were measured and both the slag and steel were sampled. The steel samples were taken through the bottom of the ladle. The injection treatment in station had been done with predetermined amount of RE. Thus RE wire (ϕ d=12mm) were injected into the ladle top with speed from 2 m/s to 3m/s and nitrogen was used as a carrier stirring gas at a pressure of 6-atm for about 6 to 10 minutes. For comparison with CaSi injection was also done into ladle top (ϕ d=11mm) at speed of 2.2 m/s. Steel samples were taken at predetermined stages, before and after stirring by gas. Samples from the rolled products were also taken. Complete chemical analysis was driven for selected samples. Samples from each heat were examined metallographically and with a microprobe analyzer by SEM, EDX and X-ray. The oxygen-gas and oxide phase were analyzed by LECO machine TC-436.

3. RESULTS AND DISCUSSIONS

3.1. Deoxidation and desulphurization in steel

The deoxidation reaction occurred in steel as follows:



Expression in acute bracket $\langle \rangle$ symbolizes for heavy floating reaction product.

The relationship between oxygen content and RE content in steel (forming RE₂O₃) is expressed by equation (2)[41] as follows:

$$\ln \text{ /O/} = -12.996 - 0.289 \ln \text{ /RE/} + 2.23 \text{ /RE/} + 0.217 \ln a_{\text{RE}_2\text{O}_3} \quad (2)$$

Optimal RE content in steel at temperature 1873K for minimum oxygen content is found to be 1.4.10⁻⁵% to 1.5.10⁻⁵%O.

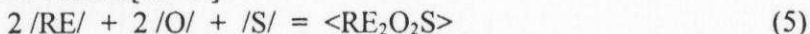
The desulphurization reaction occurred as follows:



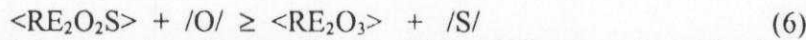
Sulphur activity in molten steel is expressed by equation (4) as follows:

$$\log a_{\text{S/}} = \log \text{ /%S/} + 0.66 - 20 \text{ /%RE/} \quad (4)$$

The RE had stronger affinity to oxygen than to sulphur, but at low temperature RE reacts easily with sulphur to form sulphides. The formed inclusion types in steel depended on sulphur content, remaining RE content and RE/S ratio. The complex oxysulphide inclusions were formed as follows[42-44]:



The formation of RE oxides, RE sulphides and RE oxysulphides is dependent on oxygen and sulphur activity. If equilibrium of reaction (5) is broken, it'll result continuous oxidation of oxysulphide phase when a part or all of RE oxysulphides are transformed into RE oxides as follows:



This sulphur goes into the steel. Thus remaining RE content in steel reduced with raising RE oxysulphides. When RE content was lower in steel, then RE oxysulphides were deoxidized and sulphur was again returned into steel, therefore it was very necessary to add aluminum before adding RE into the steel [45-48].

The aim of the RE injection into molten steel in the ladle was primarily the deoxidation. The deoxidation mainly lowered the dissolved oxygen content and changed inclusion shape control. These inclusions were easier to remove and less harmful for the mechanical properties of the steel during rolling. The results of variation of oxygen and sulphur contents in steel are shown in table 1.

Table 1. *Effect of RE additions on deoxidation and desulphurization of pipeline steel*

sample No	addition	O _{before RE}	O _{after RE}	ΔO	S _{before RE}	S _{after RE}	ΔS	RE/S	desulphurization level
-	/kg/t/	/‰/	/‰/	/‰/	/‰/	/‰/	/‰/	-	/‰/
1	CaSi 0.50	0.01774	0.01102	0.00672	0.020	0.017	0.003	-	15
2	CaSi 1.10	0.03734	0.03040	0.00754	0.014	0.016	-0.002	-	0
3	RE 0.01	0.01408	0.00510	0.00898	0.026	0.019	0.005	0.004	19
4	RE 0.02	0.01225	0.00873	0.00352	0.025	0.021	0.004	0.080	16
5	RE 0.03	0.04026	0.01440	0.00258	0.025	0.020	0.005	0.120	20
6	RE 0.05	0.01766	0.00919	0.00847	0.023	0.012	0.011	0.217	47
7	RE 0.07	0.02204	0.00926	0.01278	0.033	0.026	0.007	0.212	21
8	RE 0.10	0.01615	0.00856	0.00759	0.027	0.015	0.012	0.370	44
9	RE 0.20	-	0.01153	0.01153	0.026	0.020	0.006	0.769	23

Table 1 shows that final oxygen and sulphur contents in steel depend on their initial contents and technology of refine steel. The highest desulphurization level of 0.012%Δ_S was obtained in steel with RE addition of 0.10 kg/t (sample 8). Whilst sample with CaSi addition attained only 0.003%Δ_S. An increase in sulphur content in sample 2 addition of CaSi may be due to experimental error. The optimal ratio of RE/S in steel was 0.37. For addition of 0.10 kg/t of RE, oxygen was reduced to 0.00856%. The highest desulphurization was attained with this RE content.

Figure 1 shows when final oxygen content in steel was lower than 0.010% then it gave more efficient desulphurization of RE in steel. The final oxygen content in steel did not vary much. These results are in accordance with previous works [49-51]. Table 1 also shows that the highest reduction of oxygen content in steel was obtained from melt 7 with 0.070 kg/t RE addition by 0.0127%. Here RE/S ratio has influenced obviously on oxygen reduction (show figure 2). Figure 2 shows that the more RE/S the more is the difference of oxygen content in steel. Lower the final oxygen content in the melt, better would be the melt quality. The removal of RE inclusion was heavily influenced by the fluid dynamics of the system. This work used the nitrogen stirred ladle bottom for melts from 4 to 8. The results about oxygen

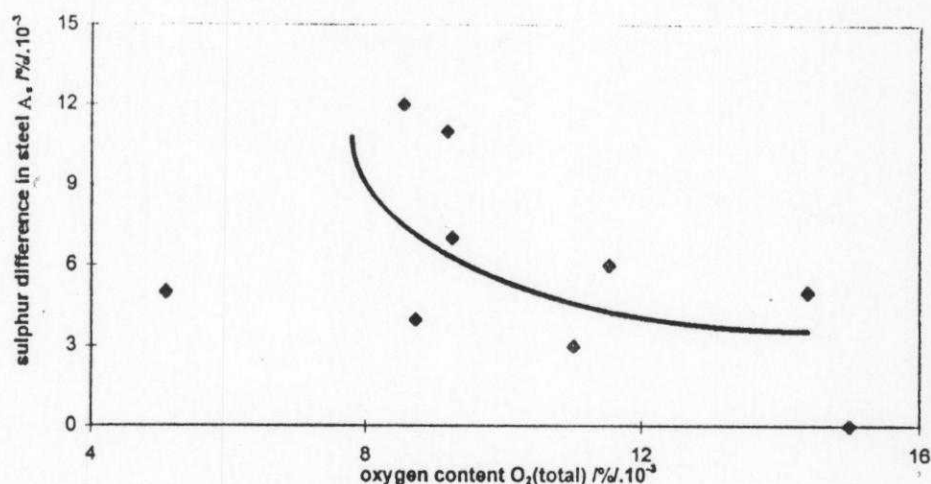


Fig. 1. Effect of final oxygen content on sulphur difference in steel containing RE

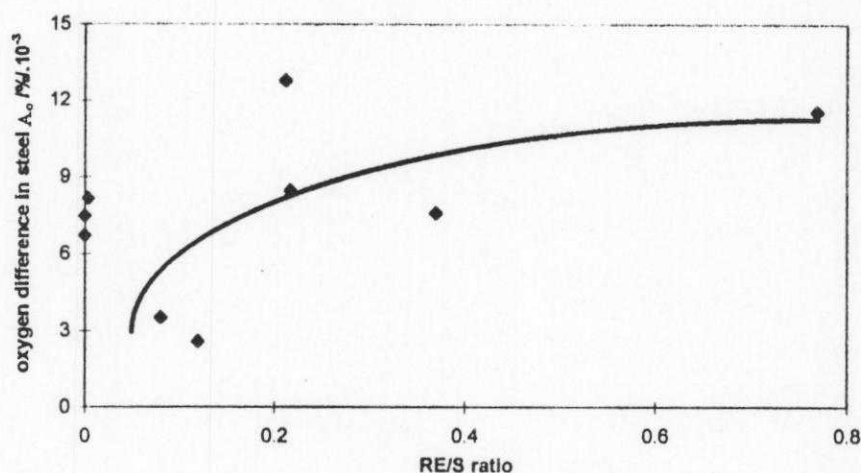


Fig. 2. Relationship between RE/S ratio and difference of oxygen content in steel containing RE

activity in steel during stirring are shown in figure 3. Figure 3 proved that the oxygen activity reduced quickly with raising RE addition. Here RE additions at 0.10 kg/t reduced oxygen activity more intensively than RE additions over 0.10 kg/t. In fact RE additions over 0.10 kg/t (shown in figure 3) significantly did not change oxygen activity. Figure 3 also shows that oxygen activity reduces faster in stirred melts than in unstirred melts.

The reduction of oxygen activity of melts containing RE and stirred by nitrogen was very obvious which resulted to the lowest oxygen content of 7 ppm (sample 9, table 2). This low oxygen content in the melt manifests itself in the form of degree of cleanliness of steel. Figure 4 shows final oxygen content in steel. When melts were not stirred by nitrogen,

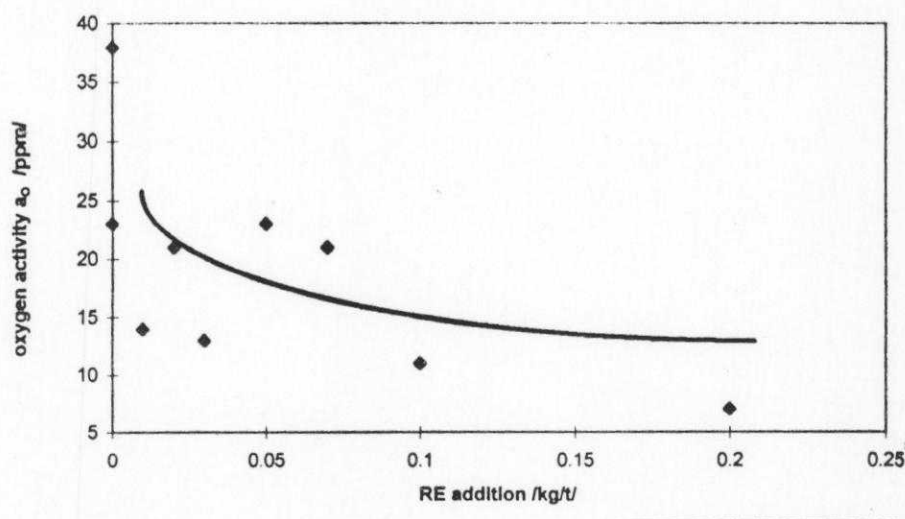


Fig. 3. Effect of RE addition and gas stirring on oxygen activity in steel oxygen content did not vary much compared to CaSi addition. The reduction of oxygen content in steel depends on stirring time and amount of RE addition (figure 4).

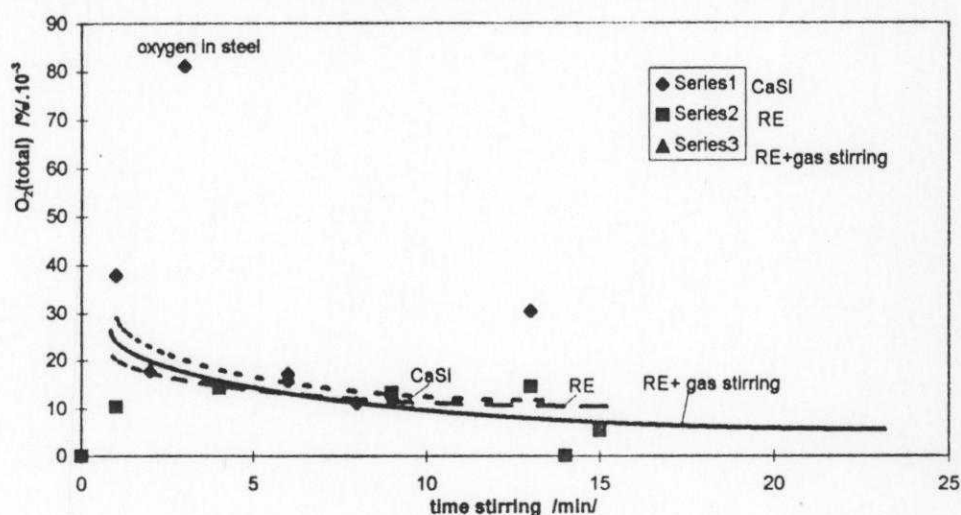


Fig. 4. Effect of time stirring and RE addition on final total oxygen content in steel

The similar to deoxidation the desulphurization also did not change, when it was not gas stirred (figure 5). Figure 5 shows that sulphur content in steel reduced faster in stirred melt than in unstirred melt. Figures 5 and 6 also show tangible estimation that the more the time of stir is the more is the oxygen and sulphur reduction in steel is. However optimum time is about 10 minutes.

The results cited above explained about the influence of gas stirring on deoxidation and desulphurization. The flotation of inclusions take place by buoyancy. Especially it has an effect on turbulent deposition and removing RE inclusions to slag. Thus lowering of the total oxygen and sulphur content can be speeded up by stirring of the melt.

3.2. Chemical composition and quantity of inclusions

The results of chemical compositions of inclusions are shown in table 3 and figures 7 and 8. Table 3 and figure 7a were show that before CaSi and RE treatment in steel oxides

Table 2. *The effect of gas stirring on oxygen content in steel*

sample No	addition RE	sampld time	stirring time	sulphur content	oxygen content	temperature	oxygen activity, a_o
	/kg/t/	/min/	/min/	/%/	/%/	/°C/	/ppm/
1	0	0	-	0.020	-	-	-
3	-	2	-	0.018	0.01774	1598	23
4	-	6	-	0.015	0.01557	1584	16
f	0	8	-	0.016	0.01102	-	-
2	0	0	-	0.014	-	-	-
1	-	1	-	0.016	0.03794	1603	38
3	-	3	-	0.016	0.08110	1607	16
4	-	6	-	0.014	0.01705	-	-
f	-	13	-	0.016	0.03040	-	-
3	0	0	-	0.026	-	-	-
2	-	4	1.45	0.021	0.01480	1606	36
4	0.01	9	-	0.020	0.01308	-	-
f	-	15	-	0.017	0.00510	-	-
4	0	0	-	0.025	-	1640	14
4	-	6	1.60	0.019	0.01225	-	-
5	0.02	9	2.00	0.022	0.01092	-	-
f	-	17	2.00	0.021	0.00873	1634	21
5	0	0	-	0.025	-	-	-
1	-	1	-	0.022	0.04026	1583	17
f	0.03	13	-	0.020	0.01440	1566	13
6	0	0	-	0.023	-	-	-
2	-	4	-	0.016	0.01766	1621	56
5	0.05	14	5.00	0.015	0.00884	-	-
6	-	21	1.80	0.016	0.01256	-	-
f	-	23	15.60	0.012	0.00919	1610	23
7	0	0	-	0.033	-	-	-
2	-	3	-	0.022	0.02204	1604	77
4	-	10	4.50	0.024	-	-	-
5	0.07	14	5.00	0.023	0.04106	-	-
f	-	20	9.60	0.025	0.00926	1577	21
8	0	0	-	0.027	-	-	-
2	-	4	-	0.015	0.01615	1614	52
4	-	8	2.30	0.016	0.01289	-	-
6	0.10	16	5.60	0.015	0.03939	-	-
f	-	18	6.00	0.015	0.00856	1600	11
9	0	0	-	0.026	-	1610	51
f	0.20	14	2.5	0.020	-	1618	7

Table 3. *Chemical composition of inclusions*

sample No	chemical content of inclusions /wt%/*					
	Al	Si	Ca	S	Mn	RE
1.1	6	21	3	37	33	0
3	40	11	3	26	20	0
4	25	2	6	45	22	0
f	27	4	5	49	16	0
2.1	17	21	4	29	29	0
3	40	0	4	38	18	0
4	21	2	17	42	20	0
f	22	3	14	40	22	0
3.2	3	8	4	46	40	0
4	10	3	4	45	38	0
f	11	3	3	45	38	0**
4.4	23	2	2	36	37	0
5	21	2	3	35	38	0
f	17	0	3	38	42	0**
5.1	9	26	25	19	20	0
f	8	2	6	48	36	0**
6.2	9	20	3	35	33	0
5	15	3	6	44	32	0
6	21	12	37	20	10	0
f	14	2	6	42	35	2
7.2	9	21	3	45	22	1
4	19	7	3	53	18	1
5	3	6	4	60	24	3
f	15	3	7	49	18	7
8.2	7	18	3	41	32	0
4	23	2	3	45	28	0
5	11	5	5	49	30	0
6	12	2	8	45	26	7
f	20	1	9	37	25	9
9.f	10	1	5	40	20	24

* oxygen content is remained

**RE content is very lower

of aluminum, manganese and silicon were stable of high temperatures e.g. $3\text{Al}_2\text{O}_3\text{SiO}_2$ (1850°C), 2MgOSiO_2 (1890°C) e.t.c.[44, 52-54]. After the CaSi and RE injection complex oxides of aluminum and RE (fig 7b) were less of $\text{REAl}_{11}\text{O}_{18}$ or REAlO_3 [42-44,53] were formed. The oxysulphides were RE and aluminum e.g. $\text{RE}_2\text{O}_2\text{S}$ and REAlO_2S [42-44,52,53] (table 4). Figure 7 also shows that separation and transformation of oxides takes place more readily (at higher temperature with lower oxygen content) with RE and CaSi addition than those without any addition.

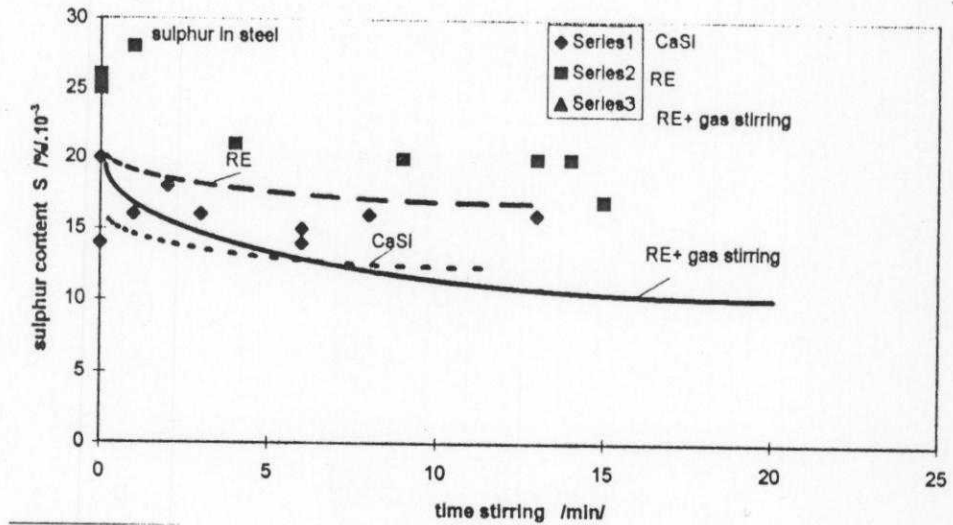


Fig. 5. Effect of time stirring and RE addition on final total sulphur content in steel

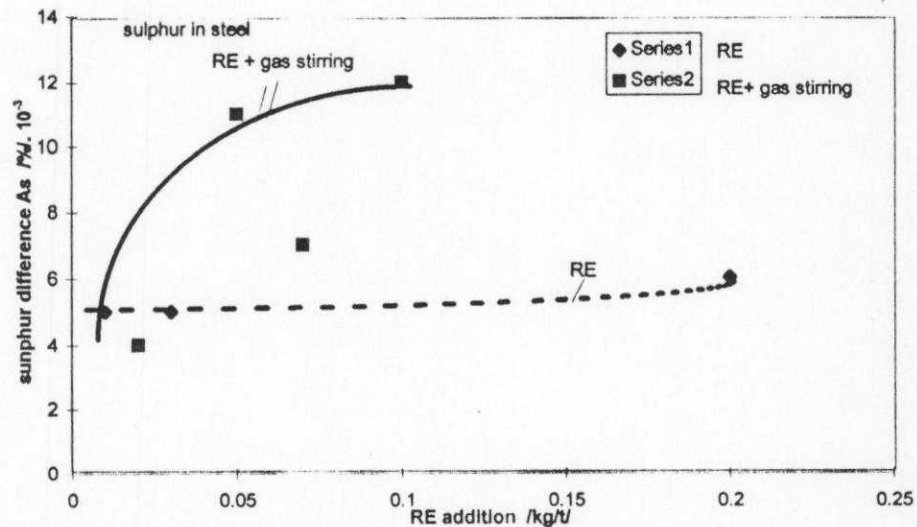


Fig. 6. Effect of gas stirring and RE addition on reduction of sulphur in steel

Figure 8 shows that before RE treatment there were inclusions having high separated temperatures of silicates (column 1900°C, figure. 8a). The RE additions with stirring by gas have helped floating part of the inclusions to slag. Remaining part of the inclusions formed complex inclusions which showed with lower oxygen content in higher separated temperature

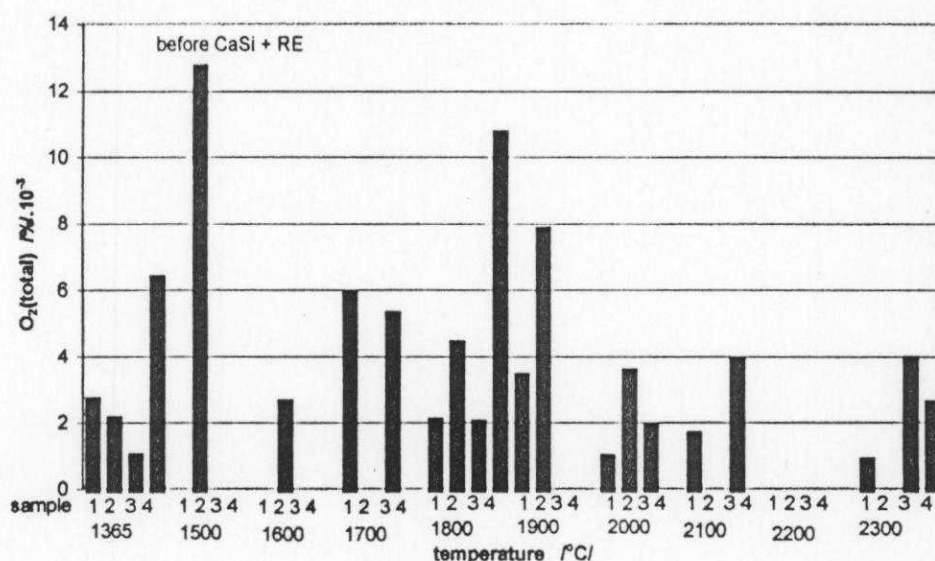


Fig. 7a. Results of phase analysis of oxides in steel on machine LECO TC 436 (for samples before CaSi and RE addition)

Table 4. Effect of RE on chemical composition of non-metallic inclusions in pipeline steel

sample No	addition RE /kg/t/	characteristic elements	prediction of inclusion composition	prediction of equations	
1	1	-	S O Mn Si Al	manganese sulphides, silicates	MnS, MnO, 3Al ₂ O ₃ , SiO ₂ ⁽⁴⁷⁾
	f	-	S O Al Mn	aluminates, manganese sulphides	Al ₂ O ₃ ,MnS, MnOAl ₂ O ₃ ⁽⁴⁷⁾
2	1	-	S O Mn Si Al	manganese sulphides, aluminates	MnO, 3Al ₂ O ₃ 3SiO ₂ , FeOAl ₂ O ₃
	f	-	S O Mn Al Ca	manganese and calcium sulphides	CaS, MnS, Al ₂ O ₃
3	2	-	S O Mn Si	manganese sulphides, silicates	MnS, MnOSiO ₂ , SiO ₂
	f	0.01	S O Mn Al	manganese sulphides, aluminates	MnS, Al ₂ O ₃
4	4	-	S O Mn Al	mangase sulphides, aluminates	MnS, Al ₂ O ₃
	f	0.02	S O Mn Al	manganese sulphides, aluminates	MnS, Al ₂ O ₃
5	1	-	S O Mn Ca Al	manganese sulphides, silicates	MnS, CaOSiO ₂ ⁽⁴⁷⁾
	f	0.03	S O Mn Al Ca	manganese sulphides, aluminates	MnS, CaOAl ₂ O ₃
6	2	-	S O Mn Si Al	manganese sulphides, silicates	MnS, 3Al ₂ O ₃ SiO ₂
	f	0.05	S O Mn Al RE Ca	complex oxysulphides RE, calcium oxides	REAlO ₂ S ⁽⁴⁷⁾ CaO
7	2	-	S O Mn Si Al	manganese sulphides, silicates	MnS, 3Al ₂ O ₃ 2SiO ₂
	f	0.07	S O RE Mn Si Ca'	complex oxysulphides RE, Al, aluminates	REAlO ₂ S, CaOAl ₂ O ₃
8	2	-	S O Mn Si Al	manganese sulphides, silicates	MnS, 3Al ₂ O ₃ SiO ₂
	f	0.10	S O RE Al Ca	oxysulphides REAl, manganese sulphides, calcium sulphides	RE ₂ O ₂ S, MnS, CaO, REAlO ₂ S
9	f	0.20	S O RE Mn Al	oxysulphides REAl, manganese and calcium sulphides	RE ₂ O ₂ S, MnS, CaO, REAlO ₂ S

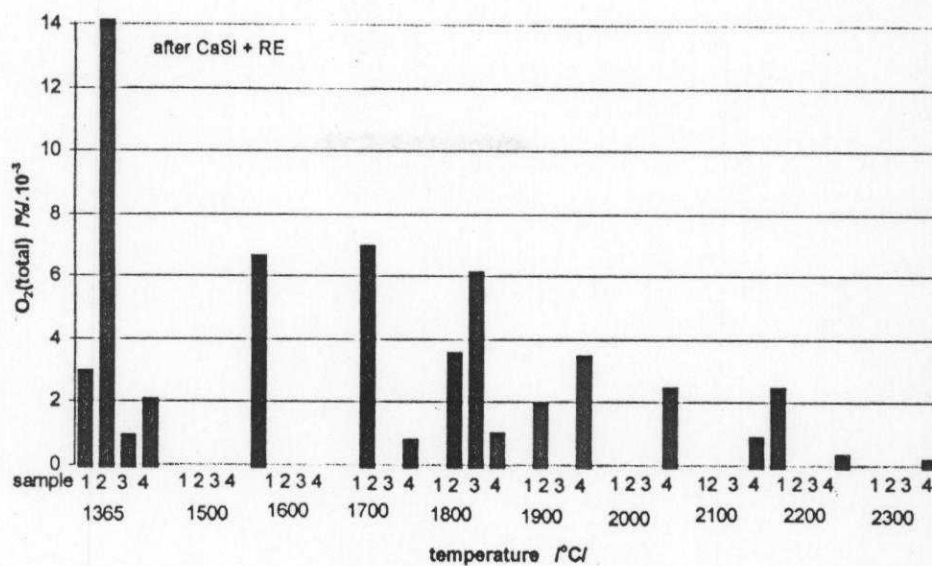


Fig. 7b. Results of phase analysis of oxides in steel on machine LECO TC 436 (for samples after CaSi and RE addition)

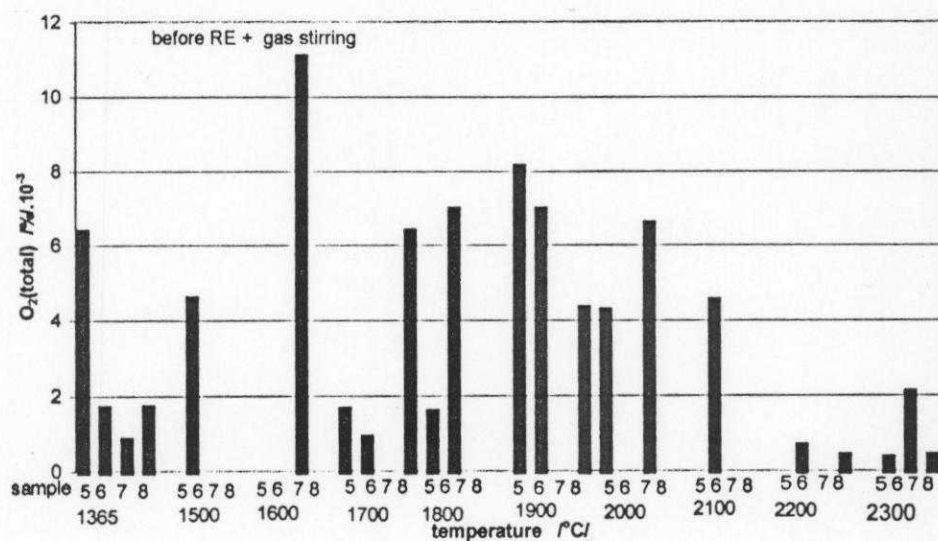


Fig. 8a. Results of phase analysis of oxides in steel on machine LECO TC 436 (for samples before RE addition and gas stirring)



(column 2000°C). After RE addition and stirring by nitrogen the number of columns of inclusions reduced from 22 to 16 and the height of columns representing oxygen content dropped below $6 \cdot 10^{-3} \%$ (fig. 8b). This is important for transformation of oxide phases in steel. This transformation is shown in figure 9. Figure 9 shows that at the beginning there were more silicates in the apex of the phase triangles and at final stage observed no inclusion in SiO_2 triangle top. These results proved obviously effect of RE injection together with gas

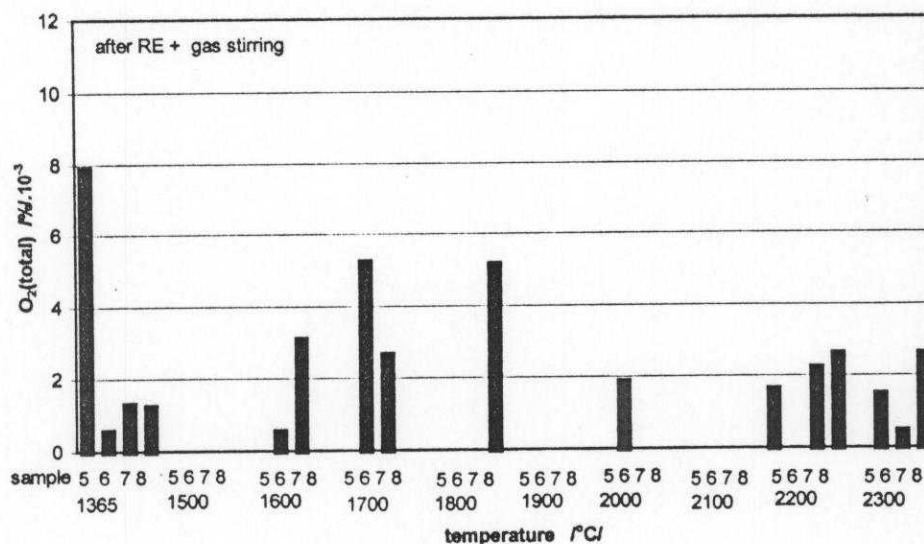


Fig. 8b. Results of phase analysis of oxides in steel on machine LECO TC 436 (for samples after RE addition and gas stirring)

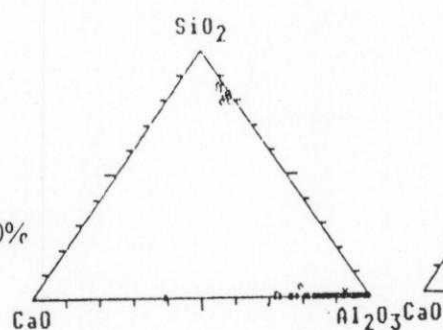
Table 5. *Quantity of inclusions*

sample No	RF addition	stirring time	inclusion number	inclusion area	inclusion number	area
	/kg/t/	/min/	-	/μm ² /	/cm ² /	/%/
1 1	-	-	263	3249	5384	0.0665
3	-	-	271	6135	6780	0.2535
4	-	-	255	1974	4417	0.0342
f	0	0	255	2071	4417	0.0411
2 1	-	-	279	6869	6282	0.1547
3	-	-	256	2156	5764	0.0486
4	-	-	291	1295	7280	0.0324
f	0	0	350	3861	5629	0.0621
3 2	-	1.45	260	2211	7318	0.0622
4	0.01	-	279	1434	6170	0.0323
f	-	-	319	1946	7183	0.0438
4 4	-	1.60	217	1831	8910	0.0589
5	0.02	2.00	274	1420	7712	0.0400
f	-	2.00	280	2138	5732	0.0438
5 1	-	-	255	870	8203	0.0280
f	0.03	0	261	1402	7346	0.0395
6 2	-	-	272	2939	6805	0.0733
5	0.05	5.00	254	1461	5199	0.0299
6	-	1.80	290	1708	8163	0.0481
f	-	15.60	269	1631	5047	0.0306
7 2	-	-	274	5016	6855	0.1255
4	-	4.50	274	2650	8814	0.0852
5	0.07	5.00	261	1182	6530	0.0296
f	-	9.60	258	2077	7262	0.0585
8 2	-	-	269	2388	7571	0.0672
4	-	2.30	270	2093	6080	0.0471
5	-	5.60	270	1226	4677	0.0212
6	-	6.00	259	1262	5832	0.0268
f	0.10	-	251	1788	3768	0.0268
9 f	0.20	2.50	272	2411	7055	0.0348

stirring on lowering of chemical composition of inclusions. These results are accordance with previous works[44,53, 54].

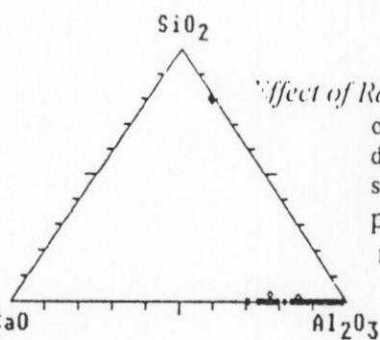
The results about inclusion quantity are shown in table 5. Table 5 shows that gas stirring reduced quantity of non-metallic inclusions in steel. Here the important criterion to estimate the content of inclusion is in terms of percentage of area of inclusions per unit surface area of microstructural sample. The lowest inclusion quantity appeared in sample 8, having 0.0268area%,(100% non-plastic): sample 6 had 0.0306 area% (100% non-plastic).

To Duy C.
oxide: 99
diluted: 2%
solid: 98%
plastic: 0%
non-plastic: 100%



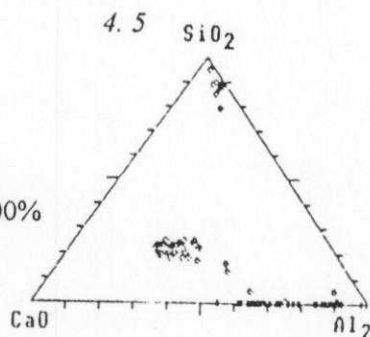
Effect of Rare Earths in steel

oxide: 85
diluted: 0%
solid: 100%
plastic: 0%
non-plastic: 100%



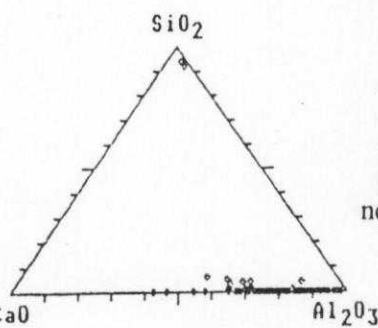
sample No 4

oxide: 155
diluted: 62%
solid: 38%
plastic: 0%
non-plastic: 100%



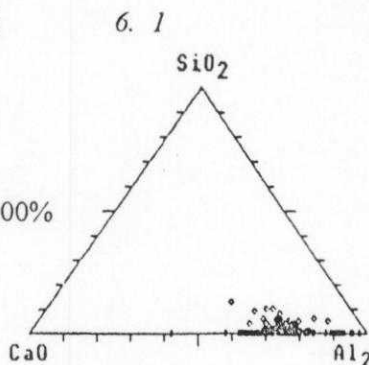
4. f

oxide: 84
diluted: 6%
solid: 94%
plastic: 0%
non-plastic: 100%



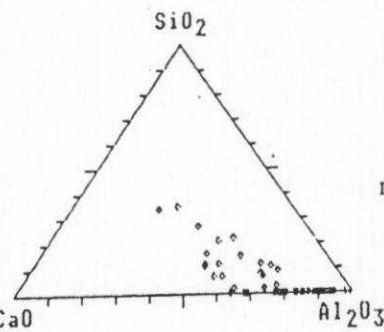
sample No 6

oxide: 111
diluted: 3%
solid: 97%
plastic: 0%
non-plastic: 100%



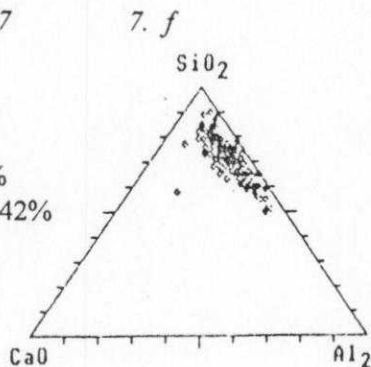
6. f

oxide: 54
diluted: 17%
solid: 83%
plastic: 3%
non-plastic: 97%



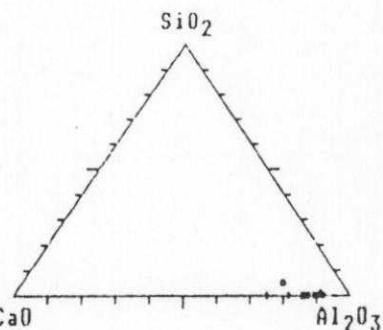
sample No 7

oxide: 114
diluted: 79%
solid: 21%
plastic: 58%
non-plastic: 42%



7. product

oxide: 19
diluted: 0%
solid: 100%
plastic: 0%
non-plastic: 100%



sample No 8

8. 2

Fig. 9. Typical effect of RE on transformation of oxide's phases in system $\text{SiO}_2\text{-CaO-Al}_2\text{O}_3$

8. product

4. CONCLUSIONS

- i) The lowest reduction of oxygen content in steel was obtained after addition RE of 0.07 kg/t. The oxygen activity was lowered to 7ppm due to addition RE of 0.2 kg/t.
- ii) The final oxygen and sulphur contents in steel depended on initial content and treatment technology of steel.
- iii) The most efficient desulphurization (from 0.027% to 0.015%S) was obtained in melt with RE addition of 0.10 kg/t together gas stirring. The desulphurization level was higher with RE addition than those with CaSi addition
- iv) The highest desulphurization was also obtained when oxygen content in steel was lower than 0.01%.
- v) Addition of RE or CaSi less than 0.03 kg/t results in the formation of inclusions containing manganese, silicates and aluminates. The oxysulphides and complex oxysulphides of RE, aluminum were formed after addition RE over 0.03 kg/t.
- vi) The all melts with gas stirring during RE additions reduced quantity of inclusions.

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