

## THE APPLICATION OF WASTE SILICA CYCLONE POWDER FOR THE PROTECTIVE COATING OF STEEL BILLETS

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The role of a protective coating is to diminish the steel surface scaling during the reheating for hot rolling. The protective coating consists of several components, and the effect of the coating is based on the formation of the modification of  $\text{Al}_2\text{O}_3$ , amorphous  $\text{SiO}_2$  and  $\text{FeO}\times\text{Al}_2\text{O}_3$ , which all exhibit low permeability to oxygen at temperature up to  $1200^\circ\text{C}$ . The silica sand powder from the cyclone is a waste product in the separation of silica sand. Tests confirmed that waste cyclone powder could replace the silica flour as one of the ingredients in the protective coating. The results of the efficiency of the protective coating after the advanced application of waste cyclone powder on AISI 1059 and AISI 6150 steels are presented. The application of the coating decreased the oxidation and decarburisation of the steel surface during the reheating for hot rolling.

**Key words:** protective coating, diminution of scaling, silica powder, use of cyclone powder

**Rabljenje odpadnog ciklonskog praha silike za zaštitnu prevlaku ljevenih gredica.** Uloga zaštitne prevlake je smanjenje oksidacije površine čelika između zagrijavanja za toplo valjanje. Zaštitna prevlaka sastoji iz više komponenti, učinak prevlake temelji se na stvaranju modifikacije  $\text{Al}_2\text{O}_3$ , amornog  $\text{SiO}_2$  i  $\text{FeO}\times\text{Al}_2\text{O}_3$ , koji su slabo propusni za kisik kod temperatura do  $1200^\circ\text{C}$ . Prah silike iz ciklona je otpadni produkt kod separacije silika pijeska. Pokazalo se, da otpadni ciklonski prah može nadomjestiti mljevenu siliku, koja je jedna od sastojaka prevlake. Prikazani su rezultati učinkovitosti prevlake na AISI 1059 i AISI 6150 čeliku. Rabljenje prevlake smanjuje oksidaciju i razugljičenje površine čelika kod zagrijavanja za toplo valjanje.

**Ključne riječi:** zaštitna prevlaka, smanjenje oksidacije, prah iz silike, rabljenje praha iz ciklona

### INTRODUCTION

During the heating of steel before hot working a layer of scale (iron oxide) grows on the surface of steel. This process is sometimes connected also with decarburisation of the steel surface [1].

Both processes decrease the yield of steel production. For this reason efforts are directed to decrease the scaling of the steel surface during the reheating process and decrease the extent of decarburisation.

The oxidation process and the oxide respectively scale formation on the surface of steel is a topic of long year investigations. It was established [2] that oxidant could cross the pore in the scale. The metal ions diffuse outwards through the layer of oxide, while the direction of the vacancies being opposite. The interruption of the resorption may induce the agglomeration of vacancies and pores appear on the oxide-metal interface. The scaling rate depends on diffu-

sion of metal ions towards the scale-gas interface however air oxygen can reach the metal surface through pores and cracks, which form because of the difference in mechanical properties and volume density of the metal and the scales. In this way, the normal composition of the surface oxide layer consisting mostly of the oxide FeO changes with faster growth of an intermediate layer of  $\text{Fe}_3\text{O}_4$  and an oxide layer of  $\text{Fe}_2\text{O}_3$ . In this reason the most efficient measure for the reduction of the intensity of oxidation is to prevent the access of oxygen to steel surface, by changing the atmosphere in the furnace or by the use of a protective coating [3] on the steel surface.

Performed laboratory and industrial tests [4 - 6] have confirmed that the use of protective coating reduces the loss of iron by scaling by 30 - 60 %, depending on the time and temperature of heating, as well as on the steel grade. The effect of protective coating is based on reactions among silicon, aluminium, iron and their oxides. At elevated temperatures amorphous  $\text{SiO}_2$ ,  $\alpha$  - modification of  $\text{Al}_2\text{O}_3$  and  $\text{FeO}\times\text{Al}_2\text{O}_3$  are formed; all compounds with low permeability for oxygen.

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By the separation of silica sand in a silica sand dig, the smallest fraction of the powder remains in the cyclones. This powder was considered a waste material and it was deposited in the dumping-ground. From ecological reasons and for reuse of waste material, the cyclone powder was applied instead of more expensive silica flour in protective coating [6]. A novel application of waste fine-grained cyclone powder of silica sand in protective coating was proposed and tested in laboratory and in industrial conditions. The aim of this investigation was to test the use of waste cyclone powder as replacement for the silica flour in the protective coating and to test the efficiency of the modified protective coating.

**EXPERIMENTAL WORK**

Thermo gravimetry (TG) and differential scanning calorimetry (DSC) tests of behaviour of modified coating were performed using the Netzsch-STA 449 C Jupiter device. Laboratory tests of reheating at different temperatures and times, as well as industrial test of protective coating were performed. The scaling and decarburisation of the steel surface was evaluated in a Nikon Microphot FXA light microscope.

**RESULTS AND DISCUSSION**

Thermo gravimetry (TG) and differential scanning calorimetry (DSC) method were performed on samples of modified protective coating and the curves in Figure 1. attained. During the reheating the first endothermic reac-

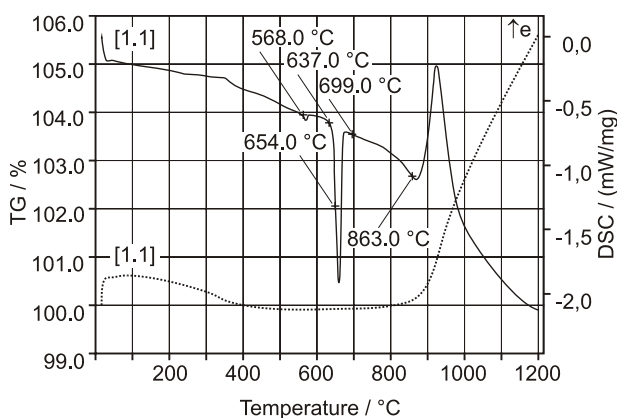


Figure 1. TG and DSC curve for the reheating of protective coating to the temperature 1200 °C  
 Slika 1. TG i DSC analiza kod zagrijavanja zaštitne prevlake do temperature 1200 °C

tions related to the degradation of the binding agent were observed. At 654 °C the melting of aluminium starts, as well as the degradation of mullite in the fireclay flour. At 863 °C a stronger exothermic reaction starts assumed to show

the oxidation of aluminium and silicon, where amorphous SiO<sub>2</sub>, FeO×Al<sub>2</sub>O<sub>3</sub>, and α - Al<sub>2</sub>O<sub>3</sub> appear, all non-permeable to oxygen.

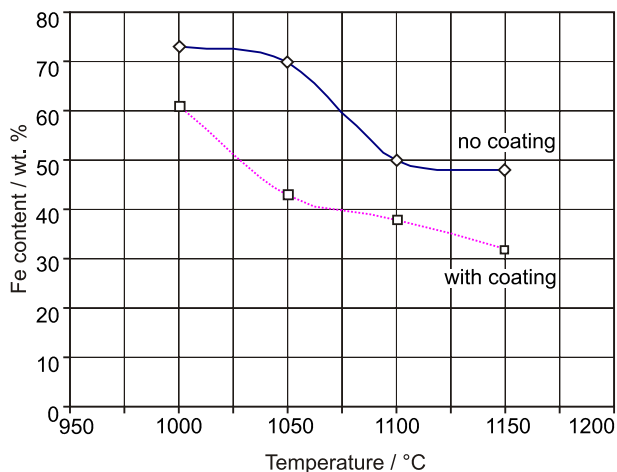


Figure 2. Content of Fe in the oxide of not coated and coated AISI 1059 steel with 0,59 wt. % C after 4 hours heating at different temperatures  
 Slika 2. Količina Fe u oksidu čelika AISI 1059 s 0,59 mas. % C, bez prevlake i s prevlakom, poslije 4 sata zagrijavanja na različitim temperaturama

Additional laboratory tests of different fine-grained ingredients for the optimisation of protective coating deposition by spraying and protective properties were performed. Tests of coating (5, 6) showed that diminution of the quantity of scale on the steel surface for up to 60 % is possible. The effect of the coating on decarburisation in industrial conditions after reheating and after hot rolling was checked with metallography of samples taken from the slabs and hot rolled bars.

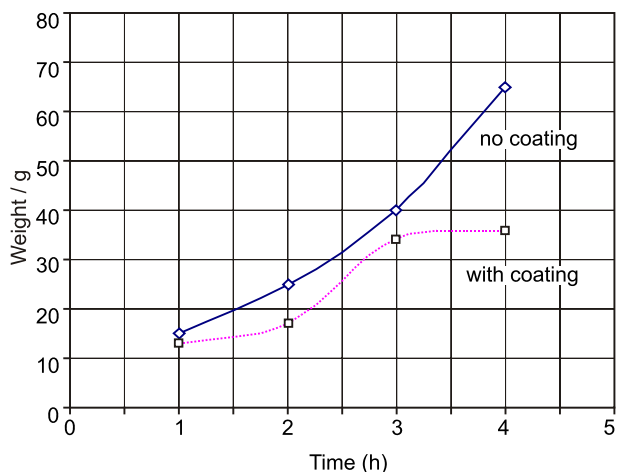


Figure 3. Comparison of the weight of oxides on not coated and coated AISI 1059 steel with 0,59 wt. % C, after 1, 2, 3 and 4 hours heating time at 1000 °C  
 Slika 3. Masa oksida na čeliku AISI 1059 s 0,59 mas. % C, bez prevlake i s prevlakom, poslije 1,2,3 i 4 sata zagrijavanja kod 1000 °C

The change of weight of samples of AISI 1059 steel (Ck 60), after heating and determination of the iron content in the oxides removed from the surface of the samples, were used for the evaluation of the coating efficiency. It was established that the coating was most effective in the temperature range from 1000 °C to 1200 °C (Figures 2. and 3.).

Laboratory tests showed at coated samples a smaller content of iron in the scale (Figure 2.) as well as decreased quantity of scale (Figure 3.). The reheating length at the laboratory tests was up to 4 hours.

The temperature range from 1000 °C to 1200 °C is appropriate for reheating before hot rolling or forging for the majority of structural steels. Higher reheating temperatures than 1200 °C are necessary only for special steels.

The modified protective coating was tested also in industrial conditions on AISI 6150 (50CrV4) spring steel (Figure 4.a, b and 5.a, b). The comparison of not coated and coated billets after reheating and after hot rolling, with

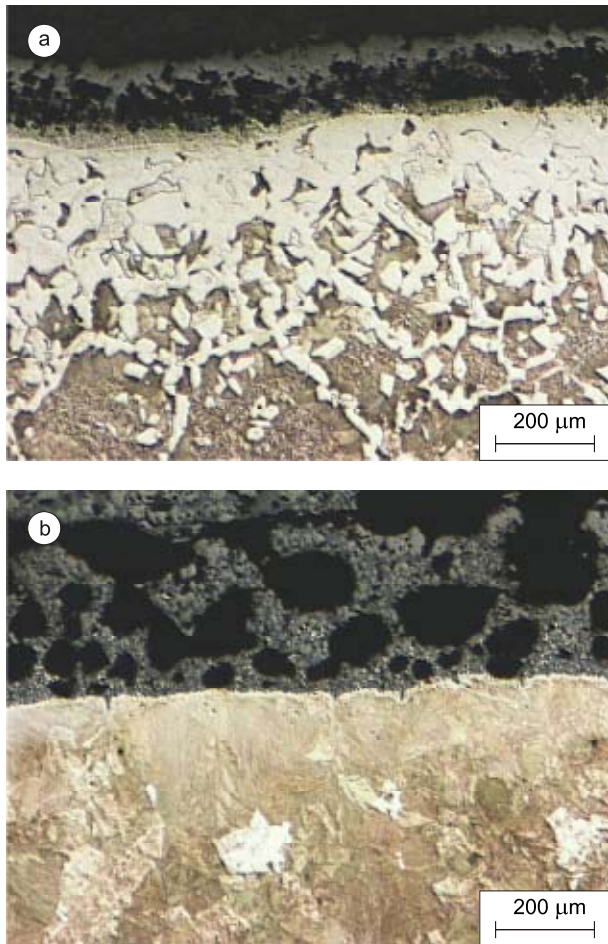


Figure 4. Microstructure of AISI 6150 (50CrV4) steel billet, after reheating for 3,5 hours at 1150 °C in a walking beam furnace: a) NVV - not coated billet, b) P II - coated billet

Slika 4. Mikrostruktura gredice čelika AISI 6150 (50CrV4), poslije zagrijavanja 3,5 sati na 1150 °C u koračnoj peći: a) NVV - gredica bez prevlake, b) PII - gredica s prevlakom

metallographical analysis on the rollings cross section, revealed that coating diminished also decarburisation of steel surface at billets as well as at hot rolled bars.

Laboratory and industrial tests confirmed that replacement of silica flour with fine grained waste silica powder collected in cyclone during the separation of natural silica sand, did not changed the efficiency of the coating. Formation of layers, low permeable for oxygen, reduces the total thickness of the oxides and also reduces decarburisation of the surface of carbon steel and spring steel.

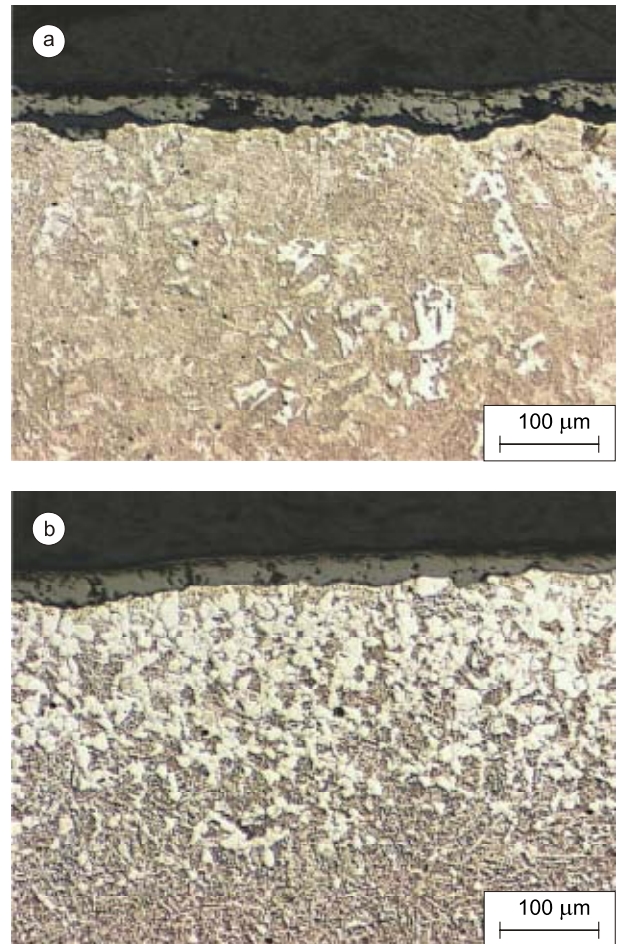


Figure 5. Microstructure of a hot rolled bar of the steel AISI 6150 (50CrV4), after reheating for 3,5 hours at 1150 °C in a walking beam furnace and rolling to 80 × 40 mm bar: a) 1 BN - billet ground and reheated, b) 2 BP - billet ground and coated before reheating

Slika 5. Mikrostruktura valjanog profila iz čelika AISI 6150 (50CrV4), poslije zagrijavanja 3,5 sati na 1150 °C u koračnoj peći i valjanju u profil 80 × 40 mm: a) 1 BN - gredica brušena, zagrijana i valjana, b) - gredica brušena i pokrivena prevlakom prije zagrijavanja i valjana

## CONCLUSIONS

1. The waste powder, a remain of the cyclone separation of natural silica sand can be used as ingredient of pro-

protective coating for decrease of the scaling of the steel surface during reheating process.

2. The performed tests confirmed that modification of protective coating, with the use of fine-grained cyclone powder, did not influence the behaviour of coating in laboratory as well as in industrial conditions, since laboratory and industrial tests confirmed the beneficial effect of the new protective coating. The decrease scaling intensity and decarburisation of carbon steel and spring steel was confirmed also. After reheating in walking beam furnace, on the surface of the billet of AISI 6150 steel without coating a decarburised layer of around 300  $\mu\text{m}$  was formed, while, no decarburisation was observed on the coated billet. Similar results were also obtained for hot rolled bars.
3. The tests also showed that cleaning of steel surface by grinding of as cast billets before reheating process accelerates decarburisation process of the steel surface.

In such a case the application of protective coating is particularly recommended.

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