OPTIMIZATION OF MATERIALS SELECTING APPLICABLE TO THERMOCHEMICAL TREATMENTS

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Abstract: This study presents a duplex treatment (aluminazing and nitrating) applied to some steels aiming at improving the properties of the superficial coating, increasing hardness and resistance against wear. Thus, there were executed 8 parts having cylindric form with a diameter of 20mm and thickness of 8mm using 15Cr8 and 40Cr10 as material and a sample with the same sizes from 39CrAl6 and 34CrAlNi7. The samples made of 15Cr8 and divided in two groups of four parts each were labelled, participating in the first group only for tincoating and the second group both for aluminazing and nitriding. The sample made of 39CrAl6 and 34CrAlNi7 was labelled and participated only in nitrating. On the samples were done visualization using the metalic graphical microscope and also micro-hardness measurements. The obtained results were eloquent confirming the possibility of modifying the superficial coatings, improving their properties.

Keywords: aluminizing, nitriding, thermical treatment.

1. INTRODUCTION

Generally, the main criterion of selecting a steel derives from the purpose and conditions of its use. There are pursuited therefore: its hardness, hardening, stability under high temperatures, tenacity, its resistance against ageing, dimensional stability, distorting, splintering possibility, etc.

The necessity of improving the surface coating of the parts, especially of the contact parts, represented a priority in the effected reasearches by the specialists on this domain. This research direction had as a goal the possibility of replacing some expensive materials with usual materials which were modified on their surface coating. The study presented further exposes few experimental tests of modifying the surface coatings, not only by methods of surfacing or surface coating, but also by using duplex treatment methods. Thus on a basic material made of OLC 45 was deposited by the laser-sintering method, a coating of Al₂O₃. At the depositing limit, in vicinity of the basic material, the resistance against ageing proved as being considerably higher than that one of the basic material itself. On those parts coated with that coating of Al₂O₃, was also applied a thermical chemical treatment of tin-coating, without being ascertained significant results.

Thus, in this study, I have proposed myself to accomplish a double thermical treatment (aluminizing followed by nitriding), which applied to some steels without aluminium to be able to offer the possibility of obtaining some features after nitrating similar to those features of the alloyed steels with about aluminium.

2. GENERAL INFORMATION

Aluminium can be an alloying element with an influence that produces surprizing effects on the hardness upon the surface coatings obtained by thermical chemical nitriding treatments, forming hard and stable nitrides even at very high temperatures.

However, when alloying aluminium (about 1...1,2%) with some steels (for instance for their improving), it is possible to appear some difficulties at the working by cutting or splintering, plastic deforming or even in case of some thermical treatments.

For removing these undesired effects of the aluminium on the features of manufacturing and of thermical treatment of the steels alloyed with this element, but using its favourable effect in case of nitriding, as a solution of solving the problem it was proposed to be used parts made by carbon steel or very low alloyed and then aluminized, diffusion annealing, adjustment and then nitrating. It is noticed that it is a complex method not usually recommended for the thermic treatment of some parts that wok in intensive working conditions (under high loadings)

3. THE EXPERIMENTAL STUDY

The description of the experimental pogramme

For developing the experimental study we have chosen two low alloyed steels for improving (STAS 791-88) namely 15Cr8 and 40Cr10 respectively 39CrAl6 and 34CrAlNi7, both alloyed with chrome in similar proportions and having nearly the same percentage of carbon in composition. When choosing those steels, it is very important the fact that one of the steels (39CrAl6 and 34CrAlNi7) contains additionally a percentage of 0,8-1,0% Al. The brand chemical composition for the two steels is shown in table 1.

Table 1. Chemical composition for the two steels

1. Chemical composition for the two steels						
Steel	15Cr8	39CrAl6	40Cr10	34CrAlNi7		
Chemical						
С	0,12-0,15	0,32-0,36	0,38-0,45	0,32-0,36		
Mn	0,60-0,90	0,55-0,80	0,60-0,90	0,55-0,80		
S	max.0,035	max.0,035	max.0,035	max.0,035		
P	max.0,035	max.0,035	max.0,035	max.0,035		
Cr	0,80-1,00	1,60-1,80	0,90-1,20	1,60-1,80		
Al	-	0,40-0,50	-	0,70-0,80		
Si	max.0,25	max.0,35	max.0,40	max.0,40		
Other	0,025	1,00	0,025	1,00		
elements						

With respect to the process developing there were manufactured 8 parts with cylindrical form with a diameter of 20 mm and a thickness of 8 mm from the material 15Cr8 and 40Cr10 and a sample with the same dimensions made of 39CrAl6 and 34CrAlNi7. To the parts made of 15Cr8 and 40Cr10 was first applied a thermical treatment of normalization $-860^{\circ}\text{C}/20 \text{ min/}$ air cooling - after that being effected the aluminization in a melted aluminium bath according to the parameters presented in table 2. Each time, after maintaining the aluminium bathing, the working samples were cooled in oil with the purpose of their annealing.

Table 2. Parameters treatment

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Sample		Aluminisation parameters					
(Notation)		T[°C]	Holding time	Cooling			
			[min]	medium			
$1.1.A_1$	$1.1.A_2$		15				
$1.1.N_1$	$1.1.N_2$						
$1.2.A_1$	1.2.A ₂	800	30				
$1.2.N_1$	$1.2.N_2$						
$2.1.A_1$	$2.1.A_2$		15	Oil			
2.1.N ₁	2.1.N ₂						
$2.2.A_1$	2.2.A ₂	850	30				
$2.2.N_1$	$2.2.N_2$						

Note: $A_1 - 15Cr8$ aluminizated; $N_1 - 15Cr8$ aluminizated and nitrided. $A_2 - 40Cr10$ aluminizated; $N_1 - 40Cr10$ aluminizated and nitrided

The sample realized of 39CrAl6 and 34CrAlNi7 was further treated thermically by a normalization - 950° C/20min/ air cooling, annealing - 900° C/20min/ oil cooling.

After these thermical treatments, the parts $1.1.N_1$, $1.2.N_1$, $2.1.N_1$, $2.2.N_1$, $1.1.N_2$, $1.2.N_2$, $2.1.N_2$, $2.2.N_2$, and the part made of 39CrAl6 and 34CrAlNi7 were nitrided.

4. DETERMINATIONS EFFECTED ON THE OBTAINED SAMPLES

After the normalization operations, aluminization and annealing, the samples $1.1.A_1$, $1.2.A_1$, $2.1.A_1$, 2.2.A1, $1.1.A_2$, $1.2.A_2$, $2.1.A_2$, $2.2.A_2$ were sectioned longitudionally and investigated using an optical metallic graphical microscope. The aspect and the size of the obtained diffusion coating was monitorized.

After the operations of aluminization and nitrading , the samples $1.1.N_1$, $1.2.N_1$, $2.1.N_1$, $2.2.N_1$, $1.1.N_2$, $1.2.N_2$, $2.1.N_2$, $2.2.N_2$ and also the sample of 39CrAl6 and 34CrAlNi7 (nitrided, too), were longitudinally sectioned. The microstructural aspects and also some hardness measurements were targeted.

After annealing, the samples were degreased and from the surface of the aluminized parts were mechanically removed the aluminium adherences.

The preparation of the samples for the microstructural analysis and for the measurements of microhardness involved gthe following operations: cleaning or degreasing, cutting off, surface grinding for becoming plate, lopishing, metallic graphical attack (nital). The samples' cutting off was mechanically accomplished and tyhe plate surface were obtained by milling with a very small advance and permanent cooling with emulsion. The abrading was accomplished with abrasive paper and water cooling and the polishing with felt (the preparation operations of the metallic graphical were realized in the laboratory). After polishing the samples were attacked with nital 5% (solution 5% nitric acid in mehyl alcohol). The microstructures were analyzed using an optical metallic graphical microscope Bel Photonics MTM also in a laboratory.

5. THE MICROSCOPIC ANALYSIS

Sample $1.1.A_{1,2}$

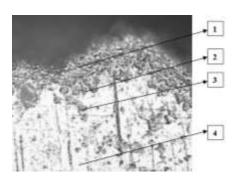


Fig.1. Microstructure of the sample $1.1.A_{1,2}$

În figure 1 is presented the microstructure of the sample $1.1.A_{1,2}$ with similar structures (normalized to $860^{\circ}\text{C}/20\text{min/air}$ cooling, annealed, mentained in a bath 15 min at the temp 800°C) with increase 400x times.

There can be observed four different elements from the surface to the part's core:

- 1-Exterior coating adhered mechanically on gthe sample's surface during the pulling out from the melted aluminium. It has the thickness of about 0,05-0,07mm. This coating isn't formed by a single compound, it contains grains of aluminium not attacked by nital and another solid intergrain constituent intensively attacked. This constituent was identified as being a form of FeAl₃ [4]. The border with the adjacent coating is labourless irregular and it is the result of the interdifusion between the two coatings.
- 2-The border separation between the surface coating (composed from aluminium and the phase of FeAl₃) and the difusion coating. It is very thin (nearly 0,01mm) and it is formed from fine particles of phase form FeAl₃ [4].
- 3-The difusion coating. It is an "alloy" (mixture) of aluminium-iron, rich in aluminium, having a thickness of 0,20-0,22mm. It is only a form compound of Fe₂Al₅ [4]. The separation surface between this coating and the basic material of the sample is irregular.
 - 4-The sample basic material, presenting a fine sorbitic structure.

Sample 1.2.A_{1,2}

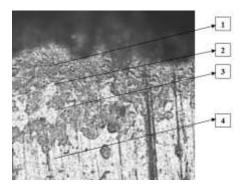


Fig.2. Microstructure of the sample 1.2.A_{1.2}

In figure 2 it is presented the microstructure of the sample $1.2.A_{1,2}$ with similar structures (normalized by $860^{\circ}\text{C}/20\text{min/air}$ cooling, annealed, bathing maintaining for 30 min at the temp 800°C), increasing 400x times. There are again noticed the four distinctive elements from the surface to the part's core, as follows:

- 1- The exterior coating mechanically adhered on the sample's surface during the pulling out from the melted aluminium bath. It has a thickness of about 0.10-0.12mm, the thickness difference copared to the sample $1.1.A_{1,2}$ due to the fast process of pulling out from the melting pot.
- 2- The separation border between the surface coating and the difusion coating is also very thin, 0,01mm. In this case it is no more that obvious presenting some "interior lines" within the difusion coating.
- 3-The difusion coating, thicker than in case of the sample $1.1.A_{1,2}$ (nearly 0,30-0,32mm) due to a prolonged maintaining. It shows the same irregular cut-out.
- 4-The basic material of the sample with fine sorbitic structure agressively attacked by nital.

*Sample 2.1.A*_{1,2}

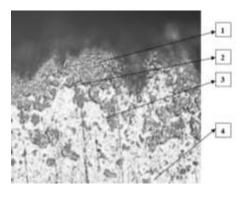


Fig.3. Microstructure of the sample $2.1.A_{1,2}$

In figure 3 it is shown the microstructure of the sample $2.1.A_{1,2}$ with similar structures (normalized by $860^{\circ}\text{C}/20\text{min}/\text{air}$, cooling, annealed, bathing maintaining for 15 min at the temp 850°C) by increasing 400x times.

- 1-The exterior coating adhered mechanically on the surface of the sample during the pulling out from the bath . Its thickness is also influenced by the speed of the pulling out. As constituents there are also aluminium and a phase form of $FeAl_3$.
- 2- The separation border of low thickness (about 0.01 mm) with a slightly sinuous aspect composed from a phase of FeAl₃.
- 3- The difusion coating with the thickness of 0,30-0,32mm , composed from a phase of Fe₂Al₅.
 - 4-The basic material of the sample with fine sorbitic structure.

Sample $2.2.A_{1.2}$

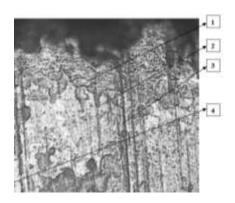


Fig.4. Microstructure of the sample $2.2.A_{1,2}$

In figure 4 it is presented the microstructure of the sample $2.2.A_{1,2}$ with similar structures (normalized by $860^{\circ}\text{C}/20\text{min/air}$ cooling, annealed, bathing maintaining for 30 min at the temp 850°C) by increasing 400x times.

- 1- The exterior coating mechanically adhered on the sample's surface during the pulling out from the melted aluminium bath. It has a thickness of the aluminium and a phase of FeAl $_3$ as compounds of about 0,1mm.
 - 2-The separation border of about 0,01mm formed from a phase of FeAl₃.
- 3-The difusion coating composed by a phase of Fe₂Al₅, with a thickness of 0,35-0,40mm due to a prolonged maintaining presenting also an irregular aspect at the separation off the basic material.
 - 4- The basic material of the sample with fine sorbitic structure

The sample $1.2.N_{1.2}$

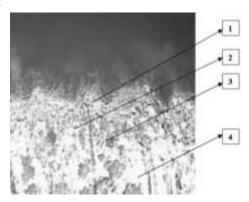


Fig.5. Microstructure of gthe sample $1.2.N_{1,2}$

In figure 5 it is presented the $\,$ microstructure of the sample $1.2.N_{1,2}$ with similar structures (normalized by $860^{o}C/20min/air$ cooling, annealed, bathing maintaining for 30 min at the temp $800^{o}C$ and nitrided) by increasing 100x times.

- 1- The exterior coating mechanically adhered on the sample's surface during the pulling out from the melted aluminium constituted from aluminium and a phase of FeAl₃.
 - 2- The very thin separation border, composed from a phase of FeAl₃.
- 3-The difusion coating composed by a phase of Fe_2Al_5 , with a thickness visible bigger (nearly 0,35-0,40 mm). The thickness increase with more than 16% of the difusion coating compared to that one measured before the nitrading is the result of the prolonged maintaining at temperatures of $560^{\circ}C$ during the nitriding. The cut-out from the basic material shows the same irregular aspect.
 - 4- The basic material of the sample with fine sorbitic structure.

*Sample 2.2.N*_{1,2}

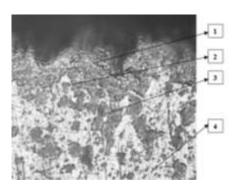


Fig.6. Microstructure of the sample $2.2.N_{1,2}$

In figure 6 it is presented the microstructure of the sample $2.2.N_{1,2}$ with similar structures (normalized by $860^{\circ}\text{C/}20\text{min/air}$ cooling, annealed, bathing maintaining for 30 min at the temp 850°C and nitrided) by increasing 100x times.

- 1- The exterior coating mechanically adhered on the sample's surface during the pulling out from the melted aluminium constituted from aluminium and a phase of FeAl₃.
 - 2- The separation border, composed from a phase of FeAl₃.
- 3-The difusion coating composed by a phase of Fe_2Al_5 , with a thickness of about 0,42-0,45mm, 20% more before the nitriding. It is the same result of the prolonged maintaining at temperatures of $560^{\circ}C$ during the nitriding. The cut-out from the basic material shows the same irregular aspect, but having a slightly finer waving.
 - 4- The basic material of the sample with fine sorbitic structure.

6. CONCLUSIONS

As a following of the aluminization, in the profile of the samples can be observed for successive zones. Thus, on the surface it was obtained a coating mechanically adhered not only during the maintaining in the melting bath, but also during the pulling out from the melting. The separation surface was irregular, in all situations, this matter being caused by the interdifusion processes which are produced during the aluminization. The aluminization temperature and also the inconstant speed of the pulling out of the sample from the melting led to a variable thickness of this first coating.

As a result of the microscopic examination it was ascertained that the exterior coating isn't constituted just from solid bound aluminium.

For the white grains of aluminium unattacked by nital, it was highlighted an intergrain constituent intensively attacked (FeAl₃).

Between the superficial coating and the difusion coating it was observed the presence of a very fine separation limit agressively attacked by nital. Then, it follows the third coating which was assimilated as being the difusion coationg, rich in aluminium and slightly attacked by nital. Consulting other specialty studies we accepted that this coating is totally composed by a form of Fe_2Al_5 . The separation between this coating and the basic material presented a very irregular aspect (similar to some "dendrites"). The difusion coating showed changes of its thickness, depending on the aluminization temperature and the duration of maintaining (from 0,20mm to 0,35mm) and the nitrided samples the thickness of the difusion zone increased to 0,42mm.

Measurements have shown that steel in the same category do not produce dramatic changes in the structure.

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