

THE INCLUSION STUDY AND THE DETERMINATION OF ALUMINUM AND INSOLUBLE CALCIUM IN AIRCRAFT STEEL SAMPLES

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Lucrarea prezintă rezultatele obținute în urma determinării părții insolubile/solubile pentru Ca și Al și studiul incluziunilor de Al și Ca în probe de oțel din aeronavă. În analiza probelor am utilizat metoda Spark-Dat, iar analiza incluziunilor am realizat-o cu Microscop Metalografic NEOPHOT 21. Valorile pentru fracțiile Al_{insol} / Al_{tot} (0,077; 0,411; 0,083; 0,097; 0,400) respectiv Ca_{insol} / Ca_{tot} (0,20; 0,14; 0,66; 0,48; 0,97), demonstrează faptul că în elaborarea oțelului au fost create condițiile pentru comportare necorespunzătoare la turnare continuă. Valorile raportului $\frac{CaO}{Al_2O_3}$ (0,00108; 0,00500; 0,02040; 0,02222; 0,18055) evidențiază faptul că oțelul din probe formează alumina solidă la temperatura oțelului topit.

The present paper presents the data obtained as a result of the determination of the soluble/insoluble part of Ca and Al and the study of Al and Ca inclusions in aircraft steel samples. When analyzing the samples, I used the Spark-Dat method and I analyzed the inclusion with the help of the 21 NEOPHOT Metallographic Microscope. The values for the fractions Al_{insol} / Al_{tot} (0,077; 0,411; 0,083; 0,097; 0,400) and Ca_{insol} / Ca_{tot} (0,20; 0,14; 0,66; 0,48; 0,97), prove the fact that in the steel elaboration, conditions have been created for an improper behavior at continuous pouring. The values of the account $\frac{CaO}{Al_2O_3}$ (0,00108; 0,00500; 0,02040; 0,02222; 0,18055) emphasize the fact that the steel from the samples forms the solid alumina at the temperatures of melted steel.

Keywords: spark diagram, inclusion, Spark-DAT, Ca insoluble, Al insoluble

1. Introduction

In the steel industry, beside manganese, silicon and calcium, the aluminum is one of the most important agents of deoxidation. During the process of micro-alloying, aluminum is used as an element of acceleration for the deep drawing improvement. Therefore aluminum may be found in steel as a metallic aluminum

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(soluble aluminum), aluminum oxide and aluminum nitride. The soluble aluminum and the nitrate one may be dissolved in acid and this is simply called, soluble aluminum. During the steel elaboration process it is important to have a measure of the level of soluble aluminum in vinegar. Between the analysis and the obtained analytical result there must be a short period of time for the application of the imposed correction. The first analysis of soluble aluminum through the spectroscopy method of optical emission was developed in the 1960s. The manganese sulphide inclusion was realized in 1961.

Aluminum alloys are characterized by their low specific weight low melting point, negligible gas solubility with the exception of hydrogen, excellent castability, good machinability, and good corrosion resistance. Premium quality castings are an essential requisite for the critical structural components used in automotive and aeronautics. The production of such castings requires that inclusions and porosity be minimized (or even eliminated) to suppress their harmful influence on the mechanical properties, so that these properties are then mainly controlled by the microstructure of the castings[1].

The presence of solid non-metallic inclusions in aluminum alloys can cause reduction in mechanical properties of castings, increased gas porosity, poor machinability, decrease in fluidity and poor surface quality. Techniques for inclusions assessment can be: quantitative metallography, chemical analysis, ultrasonic tests, SEM, EDS, OES-Spark-DAT.

The inclusions can be classified in two categories: a) total inclusions that take into account all types of inclusions existing in the cake above the filter, b) harmful inclusions, which are the sum of $Al_4C_3 > 3 \mu m$, dispersed Al_2O_3 , MgO, $MgAl_2O_4$ and potential chlorides [2].

The main inclusions that occur during melting of aluminum alloys or holding periods prior to casting are aluminum oxide (Al_2O_3) as dispersed particles or oxide films, aluminum carbide (Al_4C_3), magnesium oxide (MgO), spinel ($MgAl_2O_4$), titanium diboride (TiB_2), aluminum boride (AlB_2) and titanium aluminide ($TiAl_3$).

A great deal of effort has been expended over the years on the identification, quantification and removal of these inclusions and significant progress has been made.

In this paper my aim was to determine the concentration of Al soluble, Ca soluble, Al insoluble, Ca insoluble, through the Spark-DAT method for five aircraft samples (fuselage and support rods); the analysis of non metallic inclusion of Al and Ca for the same samples.

2. Experimental

Several qualitative and semiquantitative tests are being used by aluminum casters to estimate and thus control the inclusion concentrations in the end product.

Spark-DAT principle technique is shown in fig.1.[3]:

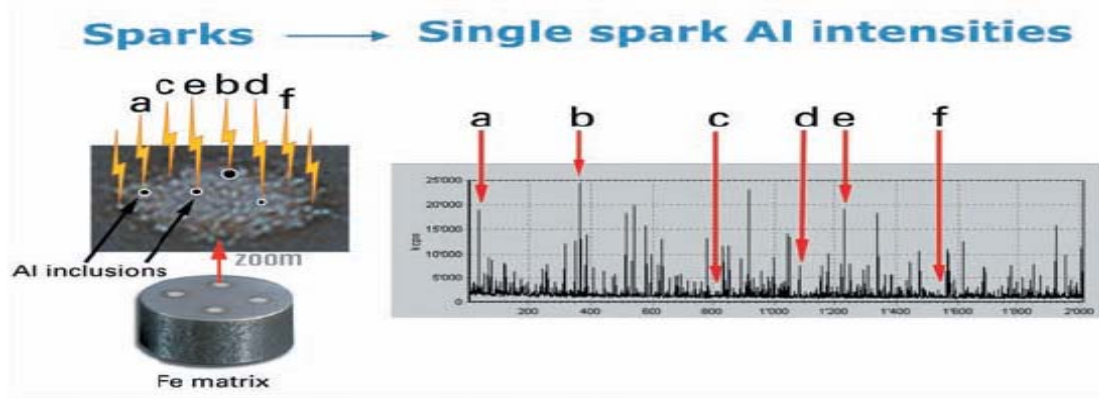


Fig.1. Principles of Spark-DAT

OES-Optical Emission Spectroscopy is the method that uses quantitative measurement of an optical emission from excited atoms to determine the concentration. Treatment and data analysis by spark (Spark Digital Acquisition and Treatment) is part of the optical emission spectroscopy method technique known largely by the speed and simplicity of determining the chemical content of metal samples.

Used very successfully in the following situations: party determining soluble / insoluble Al, B or other elements in steel; purity assessment materials, rapid assessment of the number of inclusions; rapid information on the size and composition of inclusions, contamination problems, improvement of elemental analysis.

Samples are shown in Fig. 2: S_1, S_2, S_3 fuselage of the aircraft, S_4, S_5 support rods.

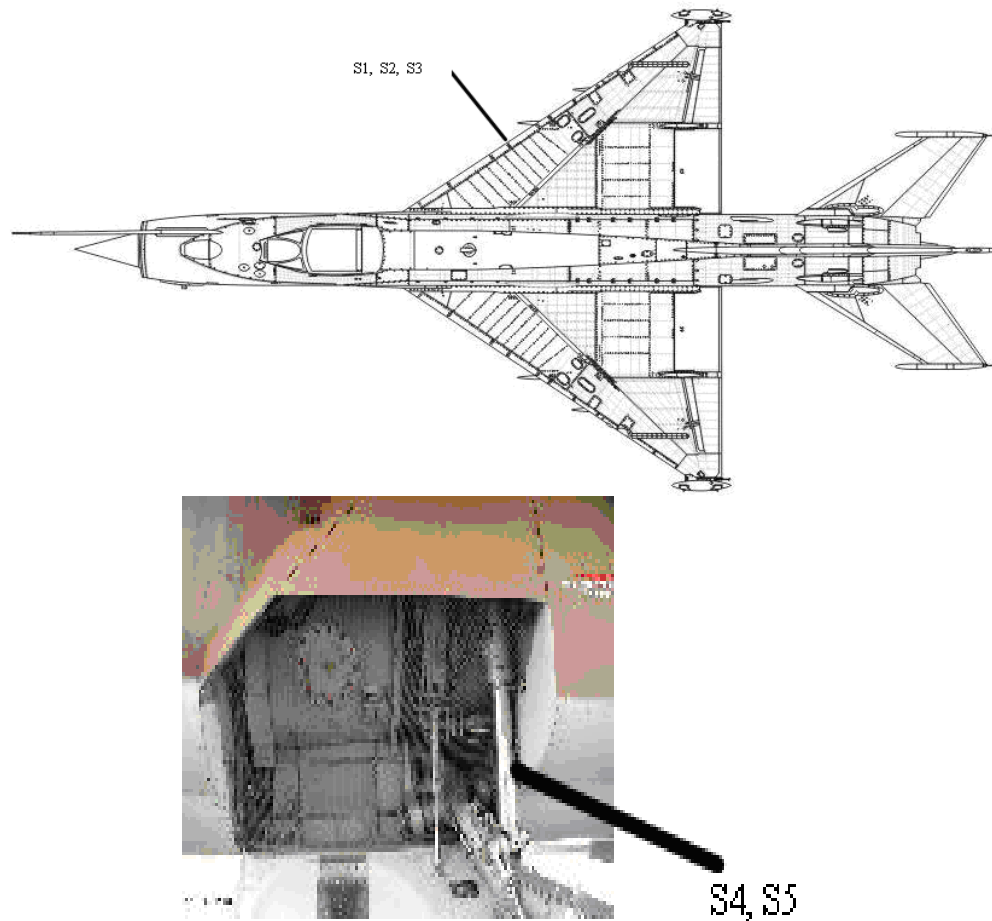


Fig. 2. Presentation of samples: S_1, S_2, S_3 fuselage of the aircraft, S_4, S_5 support rods.

The aim of the Spark-DAT option (Spark Digital Acquisition and Treatment) is to reach the intensity of each spark in the place of integrated value and to treat these signals with on-line fast algorithms. With the help of Spark DAT, the intensity emitted by the sample at each spark is measured and stored in 32 channels. The results are displayed as a spark diagram [1].

Inclusion is defined as the material with different composition (cinders, flux fondants, loam, products resulting from the chemical interaction which take place at the elaboration and pouring of the liquid metal) of the basic material, isolated in the continuous weight of the metallic material. The cast parts come from the elaboration and pouring technology, while the parts obtained through plastic deformation come from the ingot or the used semi-product [2]

The non-metallic inclusion has a strip form which produces local discontinuities in the part. These could be plastic (sulphides, silicates) and at the microscope they appear oblong, compact or fragile (oxides), which at the microscope are divided into small pieces. In all cases, the inclusion reduces considerably the plasticity and the mechanic characteristics of the metallic material.

The calcium aluminates are dangerous, especially at contact during rolling. The effect of an inclusion over the properties of fatigue is dependent of the following: the size of the inclusion, shape, heat and elastic properties and the affiliation at a matrix. These factors are related to a form factor and to the tension distribution around the inclusion. The size of an inclusion has a major effect over the resistance of fatigue [4].

Calcium is a strong deoxidant in steel, and the treatment with this chemical element can be used in the reduction of oxygen and sulphur with the alteration of the inclusion morphology. With this treatment, the expanse inclusions become globular. The aluminum oxides, which are normally of big sizes, with sharp rigs (even clusters appear), and very harmful for the devices, are in small number or are completely erased, being substituted by complex inclusion such $CaO - Al_2O_3$ or $CaO - Al_2O_3 - SiO_2$ [4].

To determine the soluble/insoluble parts, one needs the static processing of the spark diagrams. The mathematical algorithm Ca_{-tot}, Al_{-tot} calculates the medium value associated to the total account from the sample, the algorithm Ca_{-sol}, Al_{-sol} calculates the mean associated to the soluble calcium, and the algorithm Ca_{-insol}, Al_{-insol} calculates the difference between the average and the associated median with the insoluble calcium [5].

Knowing the total amount of the calcium from the sample, the relation is applied to the calibration curve[3]:

$$Ca_{insol} = \frac{Ca * Ca_{-insol}}{Ca_{-tot}} \quad (1)$$

$$Al_{insol} = \frac{Al * Al_{-insol}}{Al_{-tot}} \quad (2)$$

Sample sizes under analysis by technique Spark-DAT were $35mm \times 35mm$. The samples were polished in preparation area of the samples, using a disc grinder and abrasive paper with granulation 60.

ECRM 057, in Spectroscopic Standard Certified Reference Materials, Product no.762/ 2010 to Bureau of Analysed Samples LTD, Anglia has, been used as reference standard for steel in the five samples.

3. Results and discussion

By means of technique OES Saprk-DAT we determined elemental concentration for the samples analyzed. The table no.1 show these concentrations.

Table 1

Elemental concentration					
Concentration (ppm)/Samples	S_1	S_2	S_3	S_4	S_5
Mn (ppm)	3200	2800	3200	3100	2900
Si (ppm)	200	100	1300	300	300
P (ppm)	70	50	120	60	40
Cr (ppm)	100	400	200	200	300
Ni (ppm)	100	500	500	400	300
Cu (ppm)	100	1000	1100	1200	1200
Ti (ppm)	100	100	100	200	200
Cd (ppm)	0,8	0,5	0,9	2	1
As (ppm)	20	30	30	40	30
Al (ppm)	380	310	410	20	420

Table 2 presents results for

$$Al_{tot}, Al_{insol}, Ca_{tot}, Ca_{insol}, \frac{CaO}{Al_2O_3}, Al_{insol/tot}, Ca_{insol/tot}$$

in the aircraft samples analyzed.

Table 2

Insoluble Al and Ca in aircraft samples							
Probe	Al_{tot} (ppm)	Al_{insol} (ppm)	Al insol/tot	Ca_{tot} (ppm)	Ca_{insol} (ppm)	Ca insol/tot	$\frac{CaO}{Al_2O_3}$
S_1	440	34	0,077	0,5	0,1	0,20	0,00108
S_2	17	7	0,411	0,7	0,1	0,14	0,00500
S_3	430	36	0,083	3,0	2,0	0,66	0,02040
S_4	410	40	0,097	4,9	2,4	0,48	0,02222
S_5	20	8	0,400	4,0	3,9	0,97	0,18055

Diagram of spark for a sample (S_1) is shown in Fig. 3.

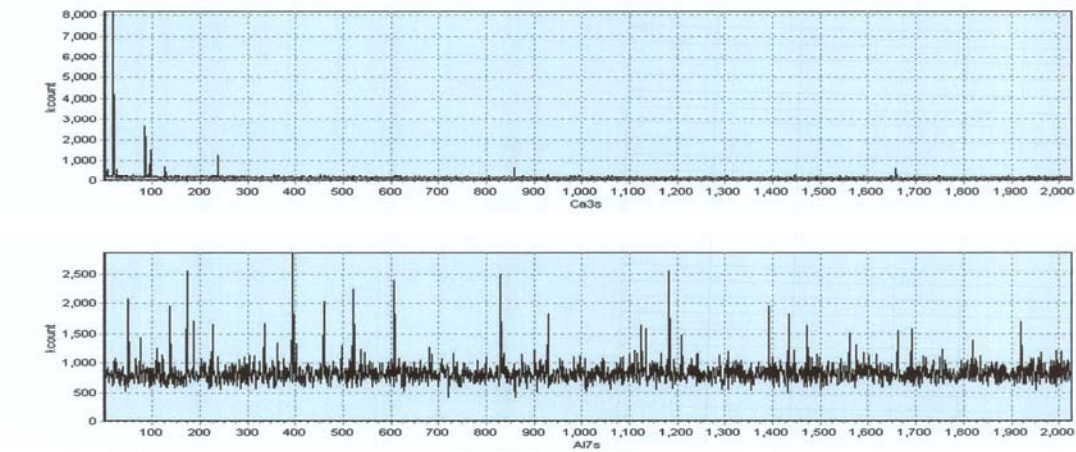


Fig.3. Spark diagram for sample 1

Fig. no.3 shows that the S1 sample has 14 Al inclusions, 5 of Ca and none of Al and Ca. The Ca inclusions are of surface, the most intense one is at spark number 20, while the Al inclusions are distributed approximately uniformly in the volume of the sample (Fig. 1). The maximum intensity for this kind of inclusion is at spark number 400.



Fig.4. Image with metallographic microscopy for sample 1 magnification of $100\times$ (globular inclusions)

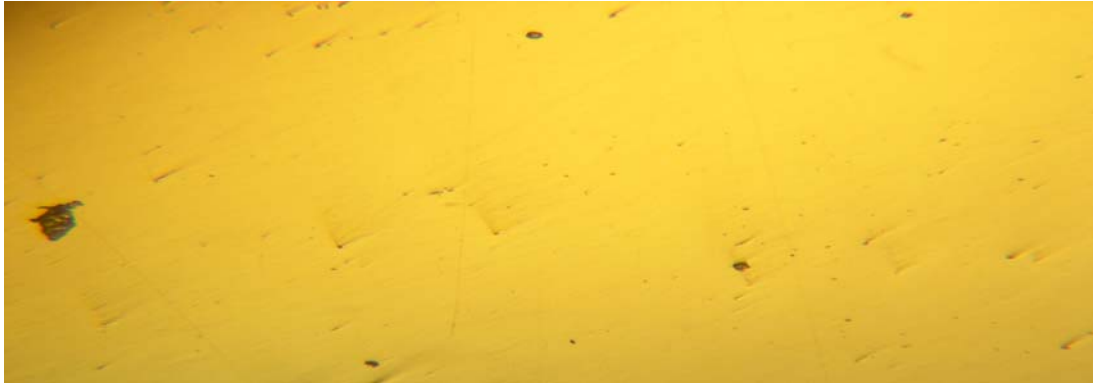


Fig.5. Image with metallographic microscopy for sample 1 magnification of 100 (irregular inclusions)

Figures 4 and 5 shows metallographic images of the disposition of calcium aluminate inclusions in samples studied, as well as inclusions: globular (Fig.4) and irregular (Fig.5).

Distribution of number of inclusions of aluminum, calcium and calcium aluminate samples fuselage and support rods, is presented in Fig. 6 diagram.

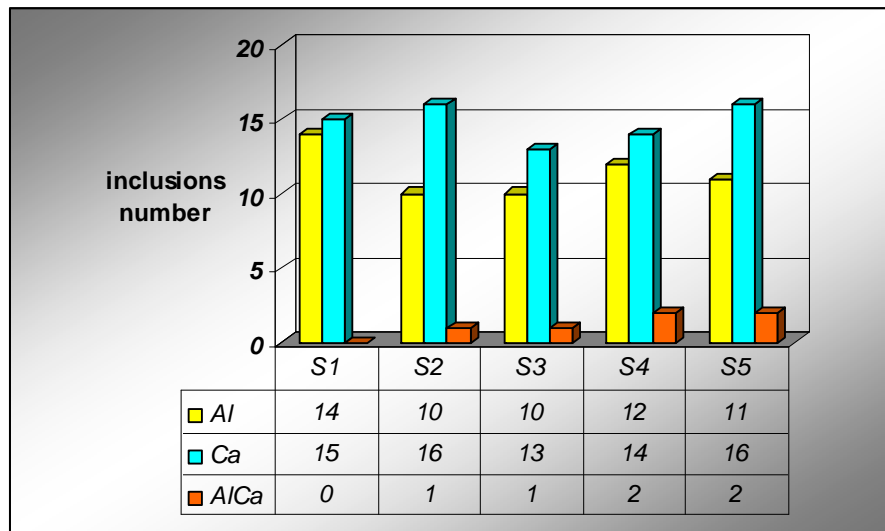


Fig. 6. The number of Al and Ca inclusions from the aircraft samples

Conclusions

The great variety of steel properties is determined by the chemical composition and the method of processing (plastic, chemical and thermochemical). The steel behavior at plastic distortion, at thermic treatment or mechanic processing is determined by mechanic composition, on the one hand, and by its method of processing, on the other hand [4].

The aim of Ca treatment is to change alumina with high heat temperatures in Al and Ca components, which are liquid at the temperatures of melted steel. The Al components which should be avoided are: $\text{CaO} \cdot 6 \text{Al}_2\text{O}_3$ with melting temperature 1833°C , and $\text{CaO} \cdot 2 \text{Al}_2\text{O}_3$ with melting temperature 1775°C . The Al component which forms $\text{CaO} \cdot \text{Al}_2\text{O}_3$ with melting temperature 1590°C , is the temperature limit for melted steel [5-6].

The Al components corresponding from the technological point of view are: $3\text{CaO} \cdot \text{Al}_2\text{O}_3$ and $12\text{CaO} \cdot 7 \text{Al}_2\text{O}_3$, and they have the following melting temperatures 1539°C , 1395°C respectively [5-6].

After applying the technique of Spark-DAT analysis on samples from the airplane fuselage and support rods may be drawn the following conclusions:

1. The $\text{CaO}/\text{Al}_2\text{O}_3$ report for these components is: 1, 63; 0, 92 respectively. Analyzing the last column from chart 1, we notice that for all these 5 samples the values of the noted report are fewer than the 2 values. We may conclude that the steel from the aircraft fuselage samples and support rods form the solid alumina at the temperatures of melted steel [5-6].
2. From the diagram analysis of the Al, Ca and AlCa inclusions, it can be noticed that number of inclusions of calcium aluminate type of aircraft is very small. This fact proves that all 5 samples are "clean" from the calcium aluminates point of view and each of them has a certain number (reduced from the technologic point of view) of Al and Ca inclusions. Knowing the distribution of Al, Ca and calcium aluminates inclusions, we may conclude over the properties of fatigue on the steel samples [5-6].
3. Spark-DAT technique can be regarded as complementary to other methods of analysis of inclusions.
4. Data from table 1 evidence reveals that the fuselage and support rods studied, concentrations of alloying elements: Mn, Si, Cu, Al, Ni, Cr steel used to classify samples as a low-alloy steel.

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