Importance of Heat Treating Procedure in the Study and the Characterization of Impurities in high alloyed Steels

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ABSTRACT

This paper deals with the methodology used in a COST 517 research concerning study of inclusions in high alloyed steel for mechanical purpose. In order to establish in a quantitative and statistically justifiable way the relationship between impurities, inclusions and mechanical properties of high alloyed steels, we have to insure that the microstructure on the whole batch of specimens of the same origin is similar. This required **appropriated heat treatments** before achieving mechanical tests. Otherwise, metallurgical properties of the samples could vary, and the relationship between inclusions and mechanical properties misunderstood.

Regardless of the melting or the atmosphere during the melting, **inclusions** are present in varying amounts in every commercial steel products. Since inclusions significantly influence properties and behavior of materials and at the same time give indications on the quality of the steel, it is quite interesting to precise **their nature and their origin**. This requires **methods of identifying** the inclusions and a knowledge of the history during steel making.

1. INTRODUCTION

To produce steel of high cleanliness with a homogeneous structure, one can use electric melting with vacuum degassing or Electro Slag Refining (ESR). Actually, in components for mechanical applications such as work rolls of cold rolling mill, the mechanical requirements (toughness, mechanical fatigue and thermal fatigue strength, etc.) involve a high rate of cleanliness of the rough material.

The roll performance in cold rolling is mostly affected by spalling, a flaw feared by rollers. Spalling can occur without any rolling incident in the last trip on the stand. A possible origin of this phenomenon is heterogeneity inside the roll due to the presence of inclusions. Spalling initiated by inclusions is extremely localized and appears like concentric rings of fatigue around inclusions. For the previous applications, the use of Electro Slag Refining could lead to a cleaner metal with smaller inclusions.

Concerning the aims of this scheme, **three goals** are to be followed up in this COST 517 research: the characterization of micro inclusions, the influence of the melting process upon the mechanical properties in service, and finally the correlation between these two parameters.

2. DESCRIPTION OF THE WORK ACHIEVED

This paper presents the methodology we put on in this COST 517 research to establish in a quantitative and statistically justifiable way the relationship between inclusions and mechanical properties. It deals particularly with the achievement of actual industrial heat treating at a laboratory level, and with some methods proposed to identify and characterize inclusions in high alloy steels.

Because of the specific origin of the rough materials, we will briefly present their chemical composition and their microstructure. After outlining the importance of heat treating, a large overview of difficulties occurring in the realization of the normalization heat treatment on the so-called MAT 1 will be given. We will lay emphasis on the size and the shape of the blocks (cylindrical part of a roll) from which samples are to be cut from, and the process finally developed.

Apart from heat treatments, it is quite important to have a look on the various inclusions founded in high alloyed steel specimens. As the study of inclusions remains difficult, we are trying to develop a systematic way of quantifying inclusions in function of their nature, based only on optical microscopy. A first stage consists of identifying inclusions by several methods: Scanning Electron Microscopy with X-rays diffraction, Interferometer Layer Microscopy. Then we try to connect this information to the optical view. The last step consists of the quantitative characterization of these inclusions in shape, size, density and distribution.

3. MATERIALS STUDIED

3.1. MORE ABOUT MELTING PROCESSES…

Some samples are obtained from ingots of the conventional melting in electric furnace process, while others samples come from the ESR process.

ESR process (fig 1) consists in remolding an electrode by means of alternative current using the Joule effect. This electrode remains submerged in a conducting slag. The main characteristic consists of a relatively short ingot mould which moves upwards whilst the consumable electrode remains stationary. As the current goes from the electrode to the ingot formed, the Joule effect heats slag.

The temperature of the slag is higher than the melting point of the steel and causes the metal to melt and flow down to the end of the electrode, where it collects in droplets and passes through the slag. Refining mainly takes place during this phase.

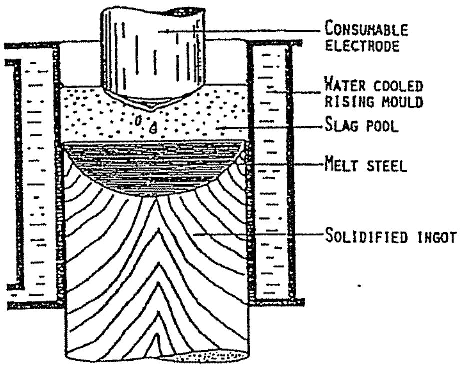

Fig 1: ESR process

The molten metal collects in the upper part of the ingot and gradually solidifies as the melting process goes on. The limit between liquid and solid metal is roughly hemispherical in shape, and this causes directional solidification of the crystals.

In comparison with the conventional electrical melting process, ESR offers several advantages:

- Greater cleanliness with elimination of large inclusions
- Reduction of undesirable elements
- Production of a sounder ingot devoid of axial porosity
- An homogeneous microstructure with uniform distribution of elements throughout the ingot
- Suppression of areas with segregation phenomenon

3.2. RAW MATERIAL

The average composition of material studied is given in table 1. High level of Cr in the content enhances the hardenability of steel alloy.

Material description	*Melting process*[1]	*C*	*Alloyed elements (% of weight)*					*V*	*Cu*
			Mn	*Si*	*Cr*	*Mo*	*Ni*		
MAT 1[2]	ESR 1 ESR 2 EF 1 EF 2	0.8 to 1.0	0.2 to 0.4	0.6 to 0.9	4.5 to 5.0	0.1 to 0.2	0.1 to 0.4	Slight traces	0.1 to 0.2

Table 1: Analysis of MAT 1

The fabrication process of rolls has a great effect on their microstructure, at the end of the casting. There are various parameters dealing with the way of conducting the casting: homogeneity of the microstructure, segregation phenomenon between core and shell, the shape and the spatial distribution of carbides... Illustrations show the microstructure of a roll coming from a conventionally electrical cast ingot (fig 2) and the microstructure of a roll of the same alloy coming from ESR process (fig 3).

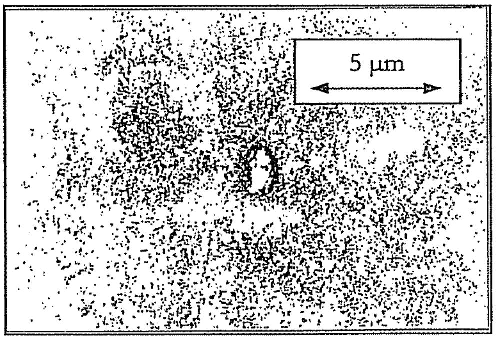

Fig 2: MAT 1 (EF) as received

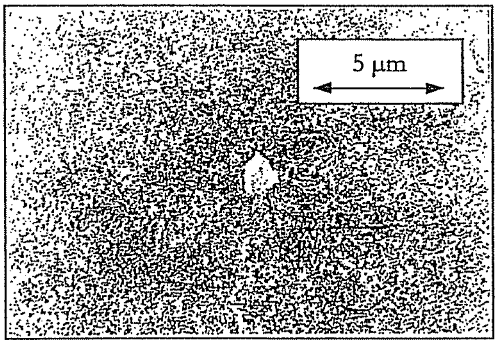

Fig 3: MAT 1 (ESR) as received

3.3. PROCEEDINGS FOR MATERIALS STUDY

Samples dedicated to a specific kind of mechanical test will be cut off from the same lump of a cylindrical part of a roll. Depending on the cost of the subsequent machining (because of the hardness of the steel after hardening), and in order to save time, heat treatments will be both done on shaped samples or blocks of steel (see the phase sequence below, fig 4). Heat treating on samples must lead to the desire microstructure and to the subsequent properties associated to the real microstructure of the roll.

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[1] For ESR, there are two different slags. With conventional electrical process (EF), there are two possibilities, depending on the presence or not of an inert gas in the stream melting.

[2] Hardened and tempered steel, for cold rolling mill rolls.

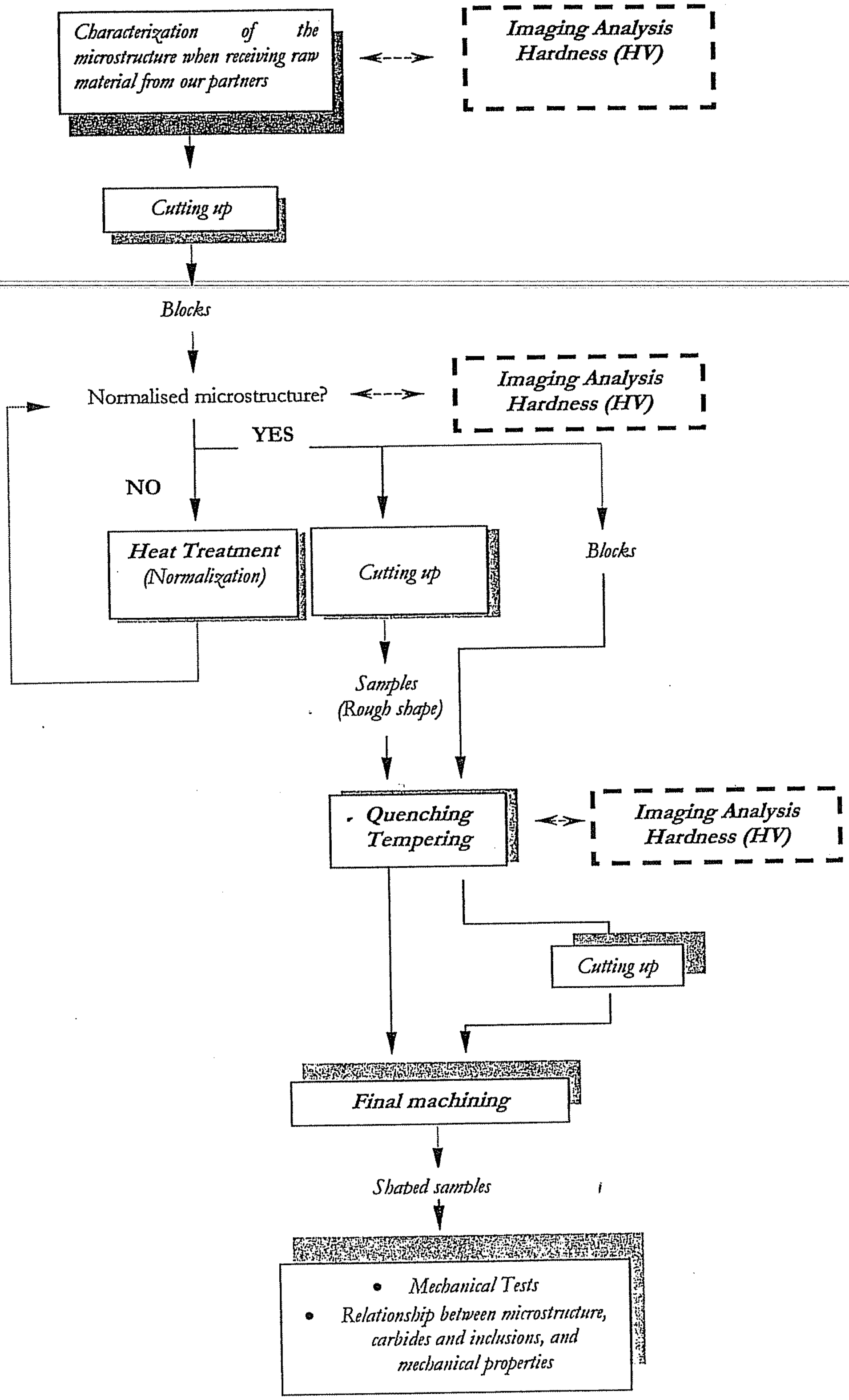

Fig 4: Methodology used in the COST 517 project

4. HEAT TREATMENTS

Although actual heat treatment is known for the given material MAT 1, a large part of experiments have been performed to find the right heat treatment to apply on each block. The heat treatment finally achieved on the block depends on his shape, his weight, and on the subsequent mechanical test achieved on samples, which are to be cut from the block. We have to take into account the fact that conditions of heat treating in industry are quite different than those been developed in laboratory.

For MAT 1 we have to perform normalization to various samples (ESR and EF) in order to achieve a similar microstructure. Hardening heat treatments (quenching and tempering) will follow this normalizing.

Only normalization heat treating on blocks of the so-called MAT 1 will be reviewed at this point, since others tests remain in study.

4.1. SHAPE OF THE BLOCKS

Cutting out of blocks on the rough material was done according to various parameters: been in effective working shell of the roll, controlling direction and number of samples, avoiding oxidation and decarburization zones due to subsequent heat treatments on the blocks, size conforming to heating power of furnace…

The weight of the blocks varies from 6 to 16 Kg, and the most commonly founded forms are illustrated on fig 5.

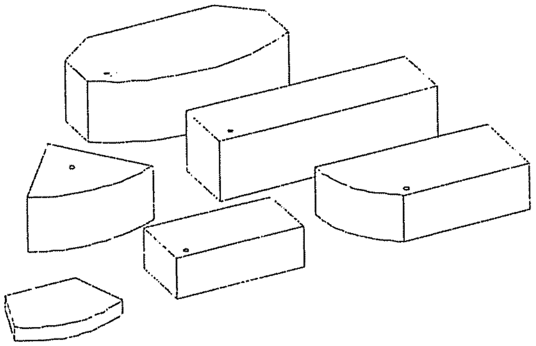

Fig 5: Shape of the blocks for the normalization heat treatment

4.2. VARIATIONS BETWEEN LABORATORY AND ACTUAL INDUSTRIAL HEAT TREATMENT

The industrial normalizing used for MAT 1 is illustrated in fig 6. But this heat treatment seems very long, as we must prepare many specimens for testing. An important part of our work consists in reducing the duration of this normalization heat treatment.

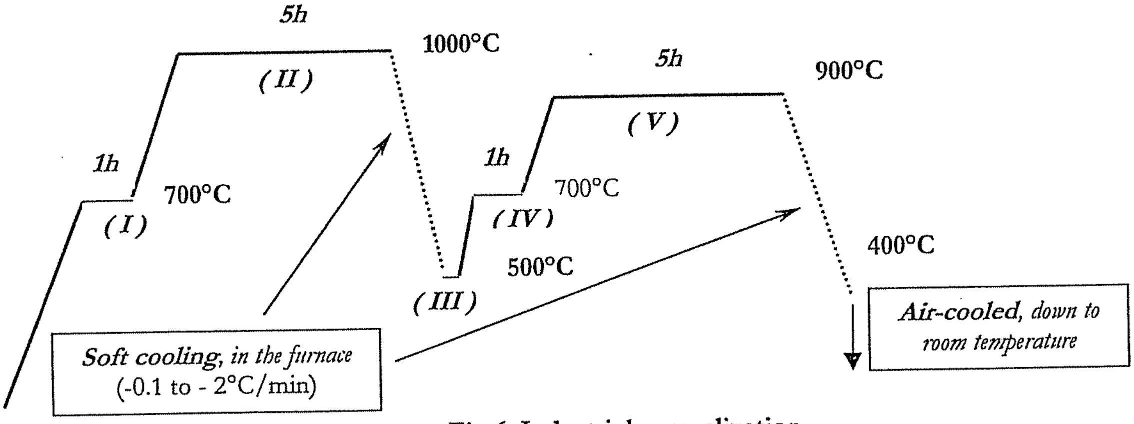

Fig 6: Industrial normalization

Many parameters have been explored while performing heat treatment: variation of the cooling rate in the mass of the blocks, influence of homogenizing prior to normalizing, influence of holding time during austenitizing [3]... The aim of these trials was to obtain the desired microstructure: **fine lamellar pearlite.**

In order to save time, the homogenizing (stage II on fig 6) MAT 1 has been suppressed, as the microstructure of rough material shows no heterogeneity.
Because of their size and the heating supply of the furnace, the heating up of heat-treated blocks remains quite uniform from the shell to the core. In consequence, stages I and IV which involve preheating for large sections, have also been cut out.
~~An intermediate cooling rate has to be realized because the microstructure obtained was not~~ suitable, when using soft cooling. During cooling, the furnace door was opened to increase the cooling rate of the current normalized block.
The holding time for the normalizing has been increased from five to six hours, to avoid the bainitic field while cooling the block. Some trials were performed with a decreasing holding time for austenitizing and a soft cooling while normalizing. These tests lead to the same microstructure as the one obtained with longer austenitizing. Hence, it may be possible to reduce further the holding time for normalization heat treatment, especially in the case of small samples.
We noticed that no more changes occur in the microstructure of the block from 600 °C, and therefore the current normalized block could be taken out of the furnace, to continue his cooling in still air. It has been noted that normalizing was not possible in still air, because of the high hardenability of MAT 1.

The modified laboratory normalizing is illustrated in fig 7.

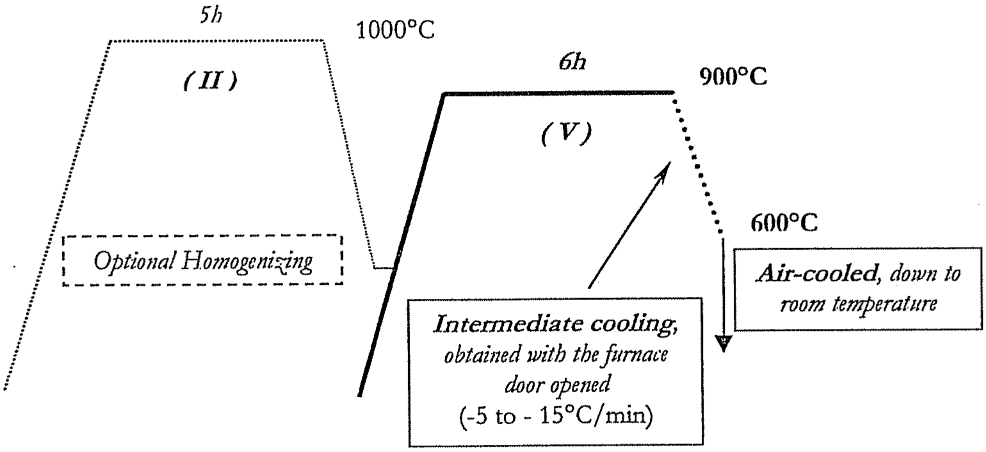

Fig 7: Laboratory normalizing

4.3. RELATIONSHIP BETWEEN HARDNESS, COOLING RATE, AND THE MICROSTRUCTURE OBTAINED.

The table II illustrates the results. The normalization heat treatment involves an austenitizing at 900°C, with a holding time of about 6h (the temperature was checked with a thermocouple put in the core of the block).
If we wish to obtain lamellar pearlite, we must have a cooling rate between 9 and 15°C/min.

Cooling rate	*Microstructure*	*Hardness (HV_{30})*
Below 0.5 °C/min	Spheroidized Pearlite	< 210 HV
Between 9 and 15 °C/min	Lamellar Pearlite	240 to 280 HV
Between 20 and 35 °C/min	Bainite	> 500 HV
Above 300 °C/sec	Martensite	> 700 HV

Table II: Results of normalizing

Beyond the previous results, many others aspects of heat treatment could appear of interest (variation of holding time and/or austenitizing temperature). As it is the rule, any increase of the holding time for a given austenitizing temperature, leads to a right shifting of the CCT curves.
According to 900°C austenitizing and 6h holding time assumption, we founded interval for the transformation points (table III).

Spheroidized Pearlite	*Lamellar Pearlite*	*Bainite**
Ar_3: 765 to 755°C	Ar_3: 745 to 730°C	Start point: 420°C
Ar_1: 745 to 730°C	Ar_1: 730 to 720°C	End point: 340°C

*Only one trial was performed

Table III: Temperatures of transformation

Variations in the values of the transformation points are due to the raw material itself, the cooling rate, the location of the block in the roll, etc.

5. CHARACTERIZATION OF INCLUSIONS

One has to analyze the micro inclusions (size from 20 µm to 200 µm) coming from the variation of some parameters of ESR and conventional electrical furnace processes. For ESR process, the emphasis will be on the chemical composition of the slag. For the conventional electrical process, the parameter to be studied is vacuum refining in ladle metallurgy, with or without any protection of the stream melting. Characterization of inclusions involves their nature, size, shape, density and distribution.
It has been noted that inclusions were most commonly present in EF samples than ESR ones and they were often tiny in shape.

5.1. MORE ABOUT THE CHARACTERIZATION OF INCLUSIONS...

Various criteria are used for the characterization of inclusions: depending on the origin, the size and distribution, or according to the malleability criterion.
Inclusion origin produces two categories: exogenous and endogenous (indigenous). Exogenous inclusions come from external sources such as refractories, while endogenous ones arise from internal sources (slag particles, other products of deoxidization or precipitation during freezing, etc.).
Inclusions may also be classified according to size: macroscopic or microscopic. But there is no clear dividing line between these two sizes. Most exogenous inclusions are generally large in size and distributed haphazardly, while indigenous inclusions are usually small. Hence, the size range

of indigenous and exogenous inclusions overlap and one cannot simply state that all indigenous inclusions are microscopic and all exogenous ones macroscopic.

The malleability of inclusions is an important parameter because it is an aid to inclusion identification, and because the shape of inclusions in wrought products has a direct influence on properties and behavior.

5.2. METHODS FOR DETECTING INCLUSIONS

Due to variation in size and distribution of inclusions, a number of procedures or methods have been developed to detect inclusions and assess their concentration [1]. We could roughly found macroscopic methods (Hot acids etch test, contact printing, fracture test, magnetic inspection, ultrasonics, etc.) microscopic ones (Optical and electron microscopy, microradiography) or chemical methods (Isolation of residues, analytical method for oxygen and sulfur). Only microscopy methods will be reviewed at this point, and especially optical and scanning electron microscopy. Besides, optical microscopy is the starting point for Charts, which allow quantitative assessment of Inclusions.

One of the most critical point of the procedure remains in the preparation of the sample prior to microscopic observation. In fact, the use of automatic polishing devices proves to be quite available to avoid scratches and staining.

After polishing, either etched or unetched samples were analyzed. Analyzing of unetched samples remains arduous, and no correlation could be done with Electron Microscopy in this state. That is the reason why we resorted to etching, which in addition revealed the nature of the matrix.

We founded difficulties in the marking of inclusions, because of their short size, in order to allow comparisons between optical microscopy and SEM. However, the final goal is a quantification of inclusions in function of their nature, based only on optical microscopy.

5.3. NATURE OF INCLUSIONS

As we said previously, we are still developing the procedure for a systematic identification of inclusions. Although all the inclusions present in the materials studied have not yet been identified, table IV gives an idea on the inclusions already met.

Fig 8 illustrates a particle founded in MAT 1-ESR. The corresponding X rays spectrum (fig 9) identified this inclusion as a Titanium sulfide.

Type of Inclusion	***Origin***	***Size***	***Malleability***[3]
Aluminum Oxide	Exogenous	Small and globular	No
Iron Alumina	Exogenous	Small and globular	No
Titanium Sulfide	Exogenous	Small and angular	No
Manganese Sulfide	Endogenous	Large and elongated	Yes
Manganese Iron Silicate	Endogenous	Small and angular	No

Table IV: Nature of inclusions founded in MAT 1

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[3] During hot working, around 1200°C

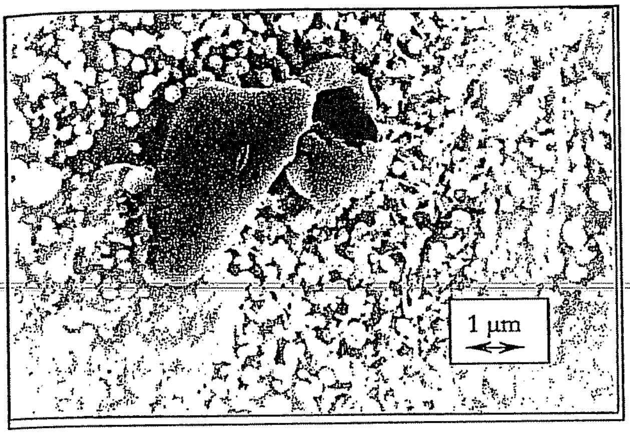

Fig 8: Inclusion of titanium sulfide in MAT 1 - ESR

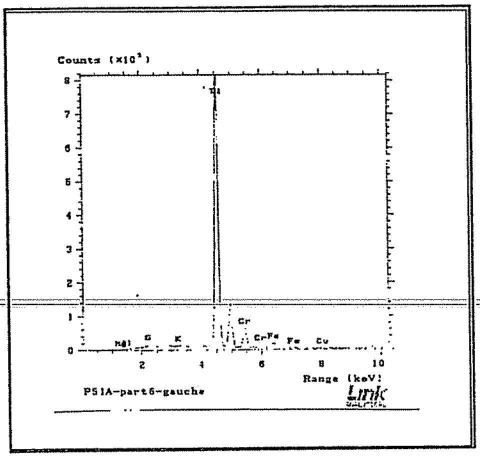

Fig 9: X-Ray analysis of inclusion of titanium sulfide

6. CONCLUSIONS

The methodology used for this study involves different steps. First we must develop an appropriated laboratory heat treatment to insure similar microstructure to each samples to be mechanically tested. For the current material (MAT 1), the desired microstructure obtained after normalizing is lamellar pearlite. We developed a laboratory normalization both to obtain this microstructure and to reduce the duration of the heat treatment. The same concept will be used for other materials to be study in the present research.

Secondly we have to analyze the inclusions present in samples. We are still developing a procedure for a systematic characterization of inclusions. The final purpose is to establish a proceeding for identifying inclusions in alloyed steels and correlate this information to optical microstructure. This microstructure will then be used to the assessment of inclusions (nature, size, shape, and distribution) by means of image analysis.

The last step will consists of mechanical tests on samples with the setting up of relationship between inclusions and mechanical properties.

For the previous reasons, it is important to insure a similar microstructure on the whole lump of samples of the same origin, and to develop a systematic procedure for the characterization of inclusions.

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