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## MULTISTAGE CRYOGENIC TREATMENT OF MATERIALS: PROCESS FUNDAMENTALS AND EXAMPLES OF APPLICATION

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### ABSTRACT

The multistage cryogenic process is an evolution from the conventional cryogenic treatments of materials. It needs shorter process time achieving the same or even better results.

This paper introduces some basic fundamentals of these treatments, their effects and applications, the equipment, etc. Some examples with different materials and from different industrial sectors are also presented as well as some brief comments about R&D and future trends of the technology.

### INTRODUCTION

People usually relates heat treatments with high temperatures, but thermal treatments can also involve cooling. Although it has been traditionally considered that deep cold temperatures have no permanent effect on the materials, it is not true at all.

Heat treatments were already known and used centuries ago but the access to really low temperatures was only possible in relatively recent days. Although the first experiences took place at the beginning of last century, it is not possible to properly speak about industrial cryogenic treatments of materials until the 70's when the liquefied gases became more affordable and the treating equipment had more accurate process control systems.

During the 80's and 90's the use of this technology increased and some treating facilities were opened, mainly in the US. Nowadays it is possible to find cryogenic processing companies in many countries all over the world.

Although still hardly known and used in Europe, the cryogenic treatment of materials is a technology that is slowly getting acceptance in industry. It basically consists in submitting the materials to deep low temperatures for increasing some of their performance characteristics like wear resistance or fatigue life.

### 1. THE PROCESS

Cryogenic treatments basically consist in submitting the materials to deep cryogenic temperatures (below 120K) following predetermined time-temperature curves in order to enhance some of their physical or structural properties.

According to the previous definition, it must be noticed that the subzero processes (at about -80 °C) that are used in many traditional heat treating facilities to reduce the austenite content in some tool steels cannot be considered cryogenic treatments.

#### 1.1 Conventional cryogenic treatments

There is no a standard process for cryogenic treatments but most of them are quite similar. In conventional cryogenic treatments the materials are slowly cooled down to a temperature

around  $-180\text{ }^{\circ}\text{C}$  and maintained for a period of time that lasts from eight hours to two or even more days. After the soak, the materials are slowly heated up to ambient temperature. Sometimes the treatment is completed with a soft tempering. The entire process typically needs two to three days to be completed.

There is a conventional process sub-category called “wet process“ where the soak is made by submersion in liquid nitrogen. Anyway, the previously described “dry process“ (no liquid nitrogen in the chamber) is more widely used.

The cryogenic treatments are performed in chambers designed for this purpose. The material is usually cooled using liquid nitrogen that is introduced in the processor through solenoid valves controlled by computer. Most of the modern chambers have heaters that also allow to control the temperature during the heating phases of the process.

## 1.2 Multistage cryogenic process

The multistage cryogenic treatment is a more advanced process that has been developed as an evolution from the conventional ones. In this treatment the isothermal soak at cryogenic temperature is substituted by several cryogenic cooling/heating phases. This process is more effective but its main advantage is that it is much faster (an average of fifteen hours for the whole process) than the conventional ones.

The cryogenic chambers that are used to apply a multistage cryogenic treatment are specially designed to perform this type of process.



**Figure 1:** Multistage cryogenic processor designed and manufactured by Cryobest International, S.L.

## 2. THEORIES AND FUNDAMENTALS

The cryogenic treatments and their applications have been developed mainly in an empiric way. This technology is becoming widely accepted and used in industry but there are still certain controversy and some mystery concerning the effects of deep cryogenic temperatures on the material microstructure. Some studies have been performed in recent years in this field and more are currently in progress all over the world.

Metallurgists know that, when a steel is quenched, usually the higher the carbon content the lower the temperature ( $M_f$ ) at which the transformation of austenite into martensite finishes. In high carbon steels, cold temperatures lead to higher contents of martensite and therefore to harder and more stable structures (more desirable in most of the applications). More recently it has also been confirmed that cryogenic temperatures promote the precipitation of fine  $\eta$ -carbide particles in the steel matrix contributing to improve the material characteristics.

These facts could explain some of the improvements in material performance due to its submission to a cryogenic treatment but, basically, they are only valid for steel. Although many grades of steel (alloyed, cold working, hot working, HSS, stainless...) can be cryogenically treated, the cryogenic processes have also evident effects in other materials like casting, cemented carbide, copper alloys, aluminium alloys, titanium, some ceramics and even certain polymers. That means that there must be something else, more general, that explains the changes in the properties of the materials.

Recent theories point to stress relieving in the microstructural level and more stable, continuous and homogeneous lattices as a key factor to achieve improved behaviours in the service performance of cryogenically processed materials. This point of view is becoming quite accepted nowadays but it must be more deeply investigated.

### **3. EFFECTS AND APPLICATIONS**

#### **3.1 Cryogenic treatment effects**

We have seen that there is a wide range of materials that can be cryogenically treated with good results. The effects of these treatments depend on the material but, in general, it is possible to obtain several of the following improvements:

- better wear resistance
- improved fatigue life
- stress relieving and dimensional stability
- increased conductivity
- improved machinability
- slight increase of hardness
- better corrosion resistance

Apart from the material, the results depend on the considered application. Of course, the cryogenic treatments are not a cure-all but there are innumerable situations where the improvements are significant or even impressive. Some examples will be commented later.

#### **3.2 Industrial applications**

It is possible to find applications in practically every industrial sector: machining, casting, injection moulding, forging, welding, automotive, aerospace, electronics, steel, timber industries, mining, agriculture, motorsports, etc. Some examples of parts that can improve their performance and increase their lives are: knives, cutting tools (drill bits, carbide inserts, mills, hobs, broaches...), saws, punches, dies, rolls, moulds, electrodes, gears, shafts, bearings, springs, cables... When a wear or fatigue problem occurs or more life is needed there is usually a good chance for using cryogenic treatments.

Cryogenic processes do not substitute conventional heat treatments although sometimes could be considered as an extension of them. One important characteristic is that they permanently affect the whole mass of the components, not just the surface. If, for example, a cutting blade is cryogenically treated, it can be sharpened as many times as desired without losing its improved performance. Another point to be taken into account is that cryogenic treatments are fully compatible with most of the surface treatments and coatings (nitriding, PVD, CVD, etc.) that are commonly used in industry to enhance the working performance of tools and components.

It is important to remark that cryogenic treatments are environmentally friendly. Absolutely no waste or residues are produced during the process. And even more, the use of this technology allows for significant reductions in energy and materials consumption.

#### 4. EXAMPLES OF APPLICATION

The number of applications of this technology is practically unlimited. Many of the most typical applications of cryogenic treatments are in the field of perishable tools. Machining tools are usually made of different grades of HSS or cemented carbide (and very often are PVD or CVD coated). These materials usually react very well to the application of a multistage cryogenic treatment. Furthermore, machining tools are often sharpened (and sometimes also coated) several times during their life. As this processes only need to be performed once this is a key advantage of this technology in many applications.

Cryogenic treatment is a good way to achieve important cost reductions and improved productivities in gear making processes. A well-known automotive supplier company can certify it. In one of the plants they manufacture steering systems for cars and trucks. The truck steering racks manufactured in this facility are machined with specially designed Maag type cutters (Fig. 3). These cutters are made of ASP2030 (powder-metallurgical HSS) and are coated with TiN every time they are sharpened.



**Figure 2:** *Maag type cutters (TRW, Spain)*

In this application the use of the multistage cryogenic treatment has made a clear difference. Without the treatment, an average of 60 parts were machined between sharpening. Nowadays all the new cutters are treated and the average production is 160 parts between sharpening. Not only this; as the wear is more homogeneous, it is only necessary to remove 0.2 – 0.3 mm. of material to sharpen the cutters (0.3 – 0.4 mm. without the treatment) what means about 30% extra uses of the tool. In this case the savings due to the use of the cryogenic processes are really important.

Another typical application of this technology is the treatment of gear cutting tools like hobs. A company specialized in the manufacturing of flywheel starter ring gears for the automotive industry uses inserted blade hobs. These hobs are made of HSS (M35) and coated with TiN. This kind of tools cannot be coated every time they are sharpened because the coating temperatures are too high for the resins that accurately fix the blades in its position. Nevertheless, the multistage cryogenic treatment avoids this problem and, in this case, the cryogenically treated hobs cut between 50 % and 100 % more ring gears than the untreated ones.

A well known aircraft manufacturer uses thousands of drill bits to make holes in difficult to machine materials like stainless steel, titanium or nickel alloys. After some months doing tests with HSS and solid carbide tools they could check that the life of the cryogenically treated drill bits was, as an average, three times longer than the untreated ones (in some cases even five times longer). Obviously, nowadays this company is achieving important tool cost savings thanks to the use of the multistage cryogenic treatment.



**Figure 3:** *Insert blade hobs after a multistage cryogenic treatment.*

Timber and wood industries as well as pulp and paper industry can also benefit from the use of multistage cryogenic treatments. Just as an example, the big knives that are used to convert the wood logs in small chips suffer severe wear. In most pulp mills these knives have to be grinded (after being previously removed) once and even twice a day. These knives are usually made of HSS and increase their life between two and three times when cryogenically treated. Taking into account that the treatment is applied only once, it is easy to understand that a growing number of companies is using this process to reduce downtime and tooling costs in their chipping facilities.



**Figure 4:** *760 mm. long wood chipping knife.*

A forging plant is using the multistage cryogenic treatment for the hot forging dies. These tools are made of hot working steel X40CrMov51 (H13). The dies are nitrided to achieve a higher hardness value in the surface. As nitriding is a surface treatment it has to be repeated after any die regrinding or restoration.

This company made tests using the multistage cryogenic treatment and the results were clear: the untreated dies made an average of 1430 parts while the cryogenically treated ones were able to make an average of 3550 (the nitrided dies have a similar performance). The next step would be to test a combination of nitriding + cryogenic but, meanwhile, the forging dies are cryogenically treated instead of nitrided.

A well known manufacturer of bearings uses steel rolls to laminate and calibrate the outer rings of the bearings. These rolls can be used to calibrate an average of 3000 units before needing grinding. The cryogenically treated rolls can make as much as 17000! units before grinding. Of course, all the rolls are now submitted to the multistage cryogenic treatment.

But not only steel or carbide tools can be bettered through the use of cryogenic treatments. Copper alloys also respond very well to the process. The resistance welding electrodes are usually made of alloys like CuCoBe or CuCrZr that have a good balance between strength and conductivity.

The influence of the multistage cryogenic treatment in the performance of resistance welding electrodes has been tested in several applications finding performance increases up to 500% in some cases. That is why nowadays there are some companies that are already using this technology to reduce their welding electrodes consumption and to increase their welding quality and productivity.

Other copper alloys like brass are also suitable for the multistage cryogenic treatment. A manufacturer of rolled profiles uses forming punches and wear parts made of brass. After two years of tests, now all the brass parts that are used in the factory are submitted to the cryogenic process. The life of those components has increased from two to three times compared with the untreated ones.

## **5. TECHNOLOGY USE AND EVOLUTION**

After some decades seeking legitimacy, it seems that the cryogenic treatments will probably become accepted and used in industry. Nowadays it is possible to find cryogenic treatment facilities in many countries all over the world, but the growth in the use of these technologies is slower than expected (the present situation of these technologies in Europe is clear example).

### **5.1 Research needs**

Cryogenic treatment fundamentals are not totally understood and this fact can explain the delay in the complete acceptance of these processes. Fortunately this situation is already changing due to the evident results of the treatments. Anyway, there is a clear need of improving the research activity in this field in order to achieve a more complete understanding of the mechanisms that are involved in the microstructural enhancement of the deep cold treated materials and also in a search of new applications.

There has been few research activity in this field of and, traditionally, most of it has been made in North America apart from some studies made in Europe. But during the last few years this situation has changed and, nowadays, there are more R&D projects concerning cryogenic treatments of materials. And, nowadays, most of the research activity takes place in Asia.

The European commission has approved the first big cooperative project in the field of cryogenic treatments. Its title is "Improvement of automotive tools and components through the application of deep cryogenic treatments". This three year project was launched in October, 2007 and the participants are research centers, universities and industrial companies from Austria, Italy, Germany and Spain. Hopefully, more new similar project proposals will also be presented in the near future in Europe.

Anyway, the empirical development of the technology will probably prevail in the development of new applications of the cryogenic treatments during the coming days.

## 5.2 Cryogenic treatments application forecast

Until now the use of this technology in industry has been mainly focused on the cryogenic treatment of all kind of tools and consumables: knives, saws, mills, inserts, dies, punches, welding tips, moulds... This is probably the most evident use for the technology because the increase of tooling life has a direct positive consequence in productivity and cost, something that is always interesting for the users.

The increase in wear resistance is the prevailing treatment effect when treating tools. But there are other treatment properties that will probably get more relevance in the coming years and one of them has special interest: the increase of fatigue life.

Probably, during the coming years there will be more and more applications where the cryogenic treatments will be used to increase the fatigue life of all kind of components. Apart from the tools the process will be applied to the manufactured components in order to increase their service life and their reliability. If this forecast is right, there will be necessary to treat much more quantities of materials. This would be a situation where the multistage cryogenic treatment has a clear advantage compared with the conventional ones: the process time is much shorter and, consequently, the treating facilities are much more productive.

### CONCLUSIONS

The treatment of materials at cryogenic temperatures is a promising and cost effective technology that, although is not new, is still hardly known and used. Only a small part of its potential has been developed.

The metallurgical fundamentals are not fully understood and the cryogenic treatments have been, and still are, developed in an empirical way. During the last years the research activity in this field has significantly increased and nowadays it is also more global than in the past.

The multistage cryogenic treatment is an evolution from the conventional ones. It is more efficient and its process time is much shorter.

The wear resistance improvement and the increase in fatigue life are just two of the effects of the cryogenic treatments. A wide range of materials can be cryogenically treated and the number of applications is unlimited. They can be found in every industrial sector.

There are two main fields of application of cryogenic treatments:

- the treatment of tools is nowadays the more common application. It is a cost effective way to increase their productivity allowing less downtime and reduced costs.
- cryogenically treating components (bearings, gears, shafts, springs...) it is possible to greatly improve their performance and reliability and also to reduce their weight and size. This field of application will probably have a big increase in the future.

The multistage cryogenic treatment is an environmentally friendly technology that helps the materials to perform better. No doubt cryogenic treatments are an exciting technology that holds much future promise.

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