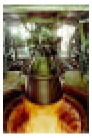

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CHARACTERISATION OF THE XD15N HIGH NITROGEN MARTENSITIC STAINLESS STEEL FOR AEROSPACE BEARING

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Abstract

The cryogenic engines of the Vulcain space launchers has been subjected to increasingly severe operating conditions so that entirely new bearings were developed for the liquid hydrogen turbopumps.

The principal axes of development, except the optimisation of the bearing geometry, were related to the improvement of the mechanical properties of the X105CrMo17 steel (AISI 440C) commonly used for this type of application and to the development of new materials able to carry out even more better performances.

A research and development program co-financed by the CNES and carried out in co-operation by SNECMA division SEP, AUBERT et DUVAL and SNR Roulements made it possible to develop the XD15NW™ nitrogen martensitic stainless steel whose properties show the possibility to operate under severe conditions of temperature, speed, loading and environment.

The XD15NW™ nitrogen martensitic stainless steel, whose advantage is to be manufactured by a classical melting process, shows interesting potentialities to replace the X105CrMo17 steel (AISI 440C) for cryogenic bearing applications with improved corrosion resistance, better metallurgical and mechanical properties, improved tribological behaviour and fatigue life.

Introduction

As part of the program for Ariane 5 new launcher of the European Space Agency (ESA), a new cryogenic hydrogen turbopump for the Vulcain engine has been developed.
In a competing technico-economic context, increasingly high levels of performance and reliability are required for the mechanisms and bearings, particularly operating under very severe conditions, so that a significant design effort has being made.
The development of the space engines with liquid propellants required the use of bearings of high technology. Indeed, the particularly hostile conditions in which they operate (high speed, significant loading and cryogenic environment) ensuring only a boundary lubrication, tend to limit the fatigue life of their components.
Materials and process were selected to meet the known or estimated criteria related to the operating conditions or the environment.
On the turbopumps of the space engines with liquid propellants (liquid hydrogen LH2 or liquid oxygen LOx) such as Vulcain, bearings are faced to very high speed conditions in a particularly severe and reactive environment (reductive in LH2 or oxydant in LOx) at very low temperature (20K for LH2 or 90K for LOx) and under boundary lubrication. Furthermore a high resistance to rolling contact fatigue as well as a great reliability are also required.

Metallic parts (rings and balls) of these bearings were traditionally made in X105CrMo17 martensitic stainless steel despite laboratory and field studies have shown its mechanical properties and corrosion resistance were weakened due to large M7C3 carbide precipitates (these carbides are preferential sites for the initiation of fatigue cracks and corrosion).

Consequently, a significant research program focused on materials and surface treatments was carried out in order to develop solutions likely to ensure a satisfying bearing performance for the required engine life. This program was carried out in close cooperation between Aubert et Duval, SNR Roulements and SNECMA division SEP.
Two topics were studied, on one hand the tribological aspect with the study of the surface damaging phenomena in relation to the means to stop them and on the other hand the relationship to basic material, particularly the influence of the manufacturing parameters and the heat treatment.
It follows that research has been directed towards the development of alloys presenting a fine and homogeneous microstructure, that led to a new family of stainless steels : high nitrogen steels (HNS) for which part of carbon was replaced by nitrogen.

The aim of this paper is to approach first the problem of the ball bearings of the liquid hydrogen turbopump by considering the investigations carried out during the programme to develop a material able to satisfy as well as possible the required performances. Work related to the development of the XD15NW high nitrogen martensitic steel is described, particularly the influence of the steel composition on the material properties (microstructure, physical and chemical properties, mechanical properties and fatigue life of bearings).

Bearings for liquid hydrogen turbopumps

Rotors for LH2 turbopumps are assembled with two pairs of angular ball bearings intended to allow the rotation of the pump at approximately 35 000rpm. They must be able to hold several times the life of the engine. For simplicity concerns and dissipated energy (several kW), these bearings operate in the fluid crossing the pump where they are installed. Indeed, the use of liquid hydrogen does not allow the use of traditional lubricants because none of them retains its properties at such low temperature. Furthermore, the liquid hydrogen viscosity is too low to ensure a significant hydrodynamic lift, so the direct contact of surfaces should be the less detrimental as possible. These bearings undergo the loads due to the transients of starting and stop as well as those due to the nominal operation of the machine, namely a few tens of daN axial and radial loads. The location of the bearings in the LH2 turbopump is shown figure 1.

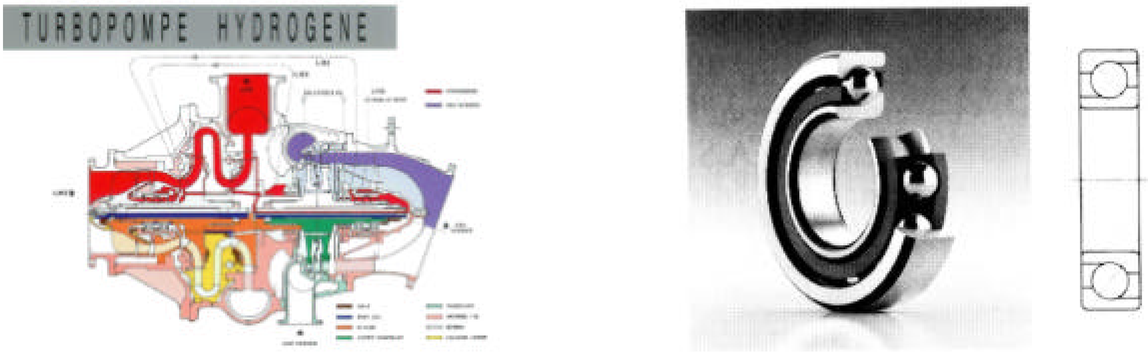


Figure 1 : LH2 turbopump and angular contact ball bearing

Following the example of many cryogenic applications having proven reliable, the initial choice for bearing materials was the martensitic stainless steel X105CrMo17.

This bearing steel is regularly manufactured by Electric Arc Furnace (EAF) then remelted by Vacuum Arc Remelting (VAR). After hardening from 1050°C, deep freezing at – 80°C and tempering at 180°C, the hardness is just above 58 HRc. The structure which results from this treatment shows a martensitic matrix and two types of carbides : micrometric and globular $M_{23}C_6$ and large eutectic M_7C_3 type carbides 10 - 40 µm in size (figures 2 and 3).

The first tests on specific bench and LH2 turbopump showed the need for modifications of the original bearings definition. These modifications supported by modelling concerned more particularly the curvature radius of the races, the contact angles, the amount and the diameter of balls in order to reduce the contact pressure. Moreover, the excessive heating and vibrations were sometime observed during tests.

Carefull investigations were carried out on the bearings of the LH2 turbopump of the Vulcain engine after operation [1]. They showed that although equipped with a ball separator containing PTFE, the transfer film of lubrication to the balls and races was insufficient. After only a few seconds in operation the contact surfaces were damaged with presence of cracks, spallings, excessive variations of sub-surface residual stresses and material transfer. The balls showed diameter variations as well as unacceptable modifications of roundness probably due to frictions (between balls and races and between balls and glass fibers of the separator) which can also explain the heat generation [2 - 3].

The same kind of damages has also been observed on LOx turbopumps bearings. A document published by NASA [4] shows that main damaging modes of the bearings of the High Pressure Oxidizer Turbo-Pump of the American Cryogenic Space Shuttle Main Engine are as follows :

- adhesive / shear pelling (ASP)
- oxidation
- abrasive wear
- fatigue (deep initiated and delamination)
- plastic deformation
- corrosion

Other papers explain the premature ASP wear by crack propagation parallel to the raceway that produce micro-debris [5] and the abrasive wear by the high friction under boundary lubrication that generate heat leading to clearance decrease and then radial pressure increase. This severe wear would also translate the insufficient wear resistance of materials.

Some basic examinations made on samples after fatigue test performed on specific benches highlight the influence of carbides on the origin of the damages.

Thus, during rotating beam tests in liquid hydrogen, L Vincent et al [6] observed broken euctectic carbides M7C3 apart from the initiation area whereas they are not broken during tests at room temperature. Two assumptions were then put forth :either an embrittlement of carbides by hydrogen or a possible weakening of the interface between M7C3 and M23C6 carbides.

During a compressive ball on plane (pin on disk machine) contact in liquid nitrogen medium V. Audrain [7] and J-L. Grosseau-Poussard [8] highlighted a degeneration of the martensite under the contact area resulting from the migration of carbon which leads to a transformation of the quadratic martensitic lattice into the cubic centered lattice of the ferrite. V. Audrain [7] showed that cracking occurs on the periphery of the residual print when the maximum tensile stress exceeds a threshold value. Moreover, V. Poupon-Claret [9] proved that the fatigue behaviour of steel is strongly influenced by the microstructure and that strain incompatibilities between M7C3 carbides and the martensitic matrix originate the fatigue cracks. Carbides are fractured and the crack propagation in the matrix is all the more fast as the hardness of this one is high. Crack growth initially in mode I then in mode II with a direction close to that of the maximum shear stress.

P. Guiraldenq et al [10] studied the effect of carbides on rolling fatigue. Their investigation showed that segregated M7C3 carbides contribute to fatigue failure while the M23C6 carbides because of their small size and of their homogeneous distribution only participate to the crack propagation. J-L. Grosseau-Poussard [8] also showed a structural change of the matrix in the cyclically stressed region under rolling plus sliding conditions on a two roller machine. Despite the steel was not damaged after 10^7 cycles under 3 GPa Hertzian stress in pure rolling conditions, even a low sliding ratio (2%) decrease drastically the performance (scuffing).

material Properties for cryogenic bearings

The basic material was studied in relation to its influence on the bearing performance, particularly the metallurgical characteristics on the physical and mechanical properties.

The functional needs are the following :

- Hertzian pressure compatibility
- Sliding and wear resistance
- Resistance to adhesion
- Surface fatigue resistance

These functions have to be satisfied in liquid nitrogen taking into account life requirements (6,000 seconds running and 20 starts).

The correspondence with the mechanical requirements are not easily quantifiable but can however be expressed as follows :

- a good subsurface rolling contact fatigue resistance
- acceptable mechanical properties at the temperature of liquid hydrogen (embrittlement risks at 20K)
- a good resistance to oxidation (condensation, possibility of storage without protective oil during several years)
- compatibility as good as possible with the temperature of surface coating processings

- an excellent dimensional stability (in relation with the presence of retained austenite)
- a fine structure free from large carbides and carbide segregation (for corrosion resistance and fatigue life)

On the basis of these requirements, the X105CrMo17 steel whose high percentage of carbon and chromium reduce the corrosion resistance and whose absence of hardening alloy elements limit the hardness proves limited.

For these reasons, a nitrogen martensitic stainless steel has been developed in such a way to improve the corrosion resistance (by reduction in carbon content, replaced by nitrogen) and the mechanical characteristics (by addition of carbide-forming alloy elements).

Nitrogen martensitic steel for bearing applications

Nitrogen, in the same way as carbon can produce either solid solution strengthening (interstitially dissolved) or precipitation hardening (nitride and carbo-nitride precipitates) of steels [11-12].

Interstitial solutes such as carbon and nitrogen exert a much greater strengthening effect than substitutional solutes. In quenched and tempered steel, nitrogen has a similar effect to carbon on the hardness of martensite, but nitrogen depresses the Ms temperature slightly less than carbon does.

Furthermore, nitrogen solubility is increased by high manganese, chromium and vanadium levels. Finally, nitrogen markedly increases the tempering resistance of high chromium steel by changing the precipitate nature while producing lower toughness that can be overcome by vanadium or niobium addition.

This type of nitrogen steel was first developed by FAG [13-14] on the basis of the X50CrMo15 steel using a special steelmaking process under high pressure (PESR steelmaking, Pressurised Electro Slag Remelting) in order to obtain a high nitrogen content (0.4%N).

This limiting aspect led AUBERT et DUVAL and SNR to develop a nitrogen steel using a more traditional steelmaking process under atmospheric pressure based on a modification of the analysis of the X65Cr13 cutlery steel. The carbon content was lowered to 0.4 %, those of Chromium was raised to 16 % together with additional contents of Molybdenum (1.75 %) and Vanadium (0.3 %), so that in these conditions the nitrogen saturation content was approximately 0. 2%.

The lower carbon content produce a smaller ratio of finer carbides compared to 440C, hence increasing the fatigue properties.

Additions of Mo and V allow a secondary hardening required for 58 HRC hardness after tempering at high temperature.

Furthermore, these elements that replace of a part of the chromium in M23 (CN)6 carbo-nitrides, formed during high temperature tempering, together with a lower carbon content should offset the chromium impoverishment of the matrix around, thus strengthening the corrosion resistance.

Consequently, the elemental composition selected after several laboratory heats for the developed steel, designated XD15NW^{TM} (type X45CrMoV15-2) is given in the table 1 :

C	N	Cr	Mo	V
0.37 - 0.45	0.16 - 0.25	15 - 16.50	1.50- 1.90	0.20 - 0.40

Table 1 : Elemental composition of the XD15NW^{TM}steel

The steel is manufactured using an electric furnace processing followed by electroslag consumable electrode remelting (E.S.R).

Nitrogen is added into the ladle by gaseous stirring and through nitrided ferroalloys. During remelting, the slag composition is selected in such a way to maintain the nitrogen content. The nitrogen content along a remelted ingot in longitudinal and transverse directions is uniformly distributed (± 0.01 % variation for 0.20 % N average level).

The lower carbon percentage leads to fine and uniformly distributed carbides. The carbide banding of XD15NW and X105CrMo17 steels are compared in figure 2. The carbide bands are notably reduced for the nitrogen steel.

Although some eutectic carbides remain, their mean size is reduced to a few µm, compared to several ten µm for 440C steel.

Figure 2 : Banding carbide structure of XD15NW and X105CrMo17

Metallurgical and mechanical properties at room temperature

The mechanical properties have been optimized in order to simultaneously obtain the following properties :

- hardness ≥ 58 HRC
- retained austenite ≤ 10 %

Several heat treatments have been performed to provide adequate quench-hardening. The influence of austenitizing and tempering temperatures on the hardness and the retained austenite for a XD15NW steel with 0.65 % C+N content is shown in figure 4.

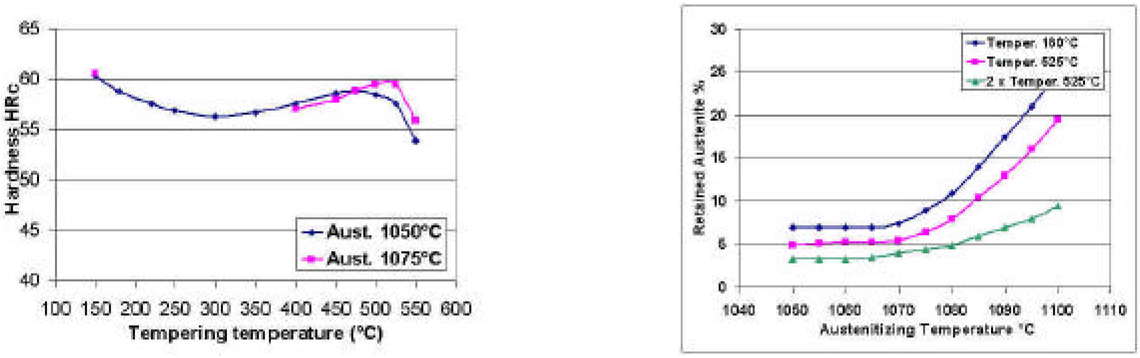


Figure 4 : Effect of heat treatment conditions on hardness and retained austenite

After quenching in oil from 1050°C, deep freeze and tempered at low temperature, the hardness is ≥ 58 HRC over a depth of 50 mm. Deep freeze to - 80°C lowers the retained austenite and raises the hardness to 60 HRC after tempering between 150 and 200°C. A second cooling does not improve this value.

When increasing the tempering temperature, the hardness drops and then a secondary hardening occurs. The beneficial effect of nitrogen is retained at all temperature. It results in higher hardness obtained with higher tempering temperature. A better hardness retention is also observed after high temperature ageing.

A second tempering at high temperature decreases the amount of retained austenite and increases the hardness.

On the basis of these results, two heat treatments complied with the required properties :

- Low temperature tempering treatment : austenitizing 1050°C, deep-freezing -80°C, tempering 180°C.
- High temperature tempering treatment : austenitizing 1075°C, deep-freezing -80°C, double tempering 500°C.

SEM (Scanning Electron Microscopy), TEM (Transmission Electron Microscopy) examinations and EDX analysis (Energy Dispersive X-rays) has been made on the structure resulting from the selected treatments.
The microstructure achieved after a low temperature tempering for XD15NW and X105CrMo17 are illustrated in figure 3. It reveals for XD15NW steel a fine martensitic micro-structure containing a fraction of about 5 % of intergranular and intragranular precipitates, insulated or agglomerated, identified as M23C6 type carbides containing in average 4 to 5 % Mo, without nitrogen [8].

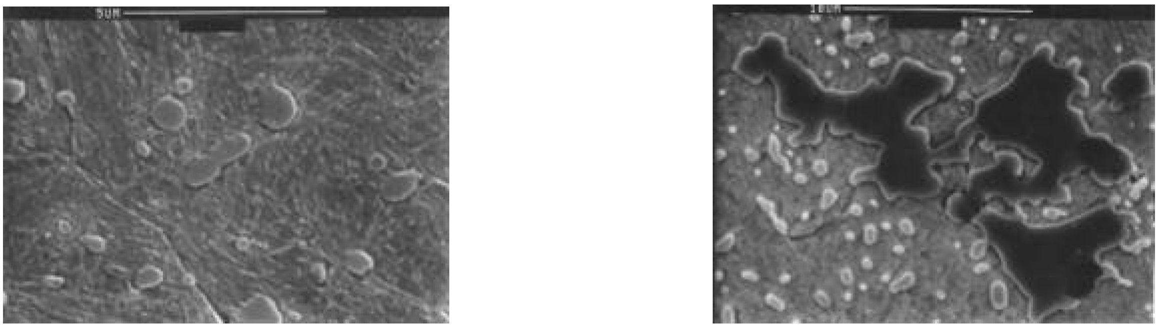


Figure 3 : SEM picture of carbides in XD15N^{TM} and in X105CrMo17 (440C)

After high temperature tempering, the microstructure is quite the same except that nitrides nano-precipitates (VC-VN-CrN) a few nm in size has been identified.

Some additional heat treatments were carried out in order to optimise the resistance and the toughness for subsequent induction hardening.
A toughness in the range of 100 MPa√m could be obtained after high temperature tempering at about 725-750°C that leads to 33 – 37 HRc hardness.
The following characteristics could be achieved in the induction hardened layer :

- Surface hardness 750-800 HV, 1.7-1.8 mm conventional depth at HV 550 after a low temperature tempering.
- Surface hardness 700-720 HV, 1.45-1.55 mm conventional depth at HV 550 after high temperature tempering.

Corrosion resistance

Three types of tests were carried out in order to evaluate the corrosion resistance.

- Salt spray test according to NFX 41.002 standard
- 98 % relative humidity test at 40°C during 2200 hours
- Current density-potential measurements in 1 % H_2S0_4 acid-aqueous solution.

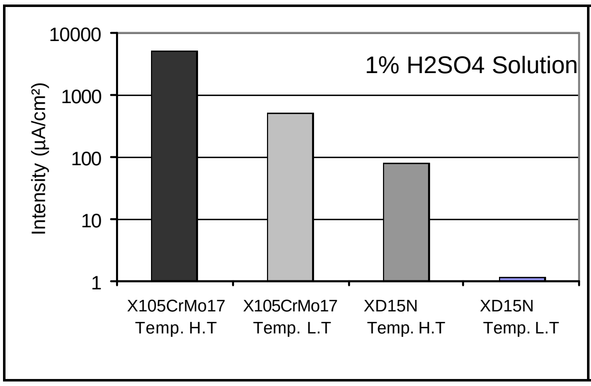
The results concerning the atmospheric tests (table 2) representative of pitting corrosion resistance are expressed by comparison to a standardised scale between 10 (no corrosion) and 0 (100 % of the surface corroded).

Test	XD15N		X105CrMo17 (440C)	
	Tempered 180°C	Tempered 525°C	Tempered 180°C	Tempered 480°C
Salt spray 24 h	10	6	7	2
96 h	7	3	4	1
264 h	7	3	2	0
Relative humidity after 2200 h	9	8	9	8

Table 3 -- Results of atmospheric corrosion tests

The current density-potential test representative of the uniform corrosion refers to the critical current density of the come-back peak that is characteristic of the material depassivation. The results are shown in figure 4.

In all cases low tempering treatment shows better corrosion resistance than the high tempering treatment, due to the carbo-nitride precipitation that occurs above 400°C during the later one. Furthermore, if we consider equivalent tempering conditions, the corrosion resistance of XD15NW is better than for the X105CrMo17 used as a reference.



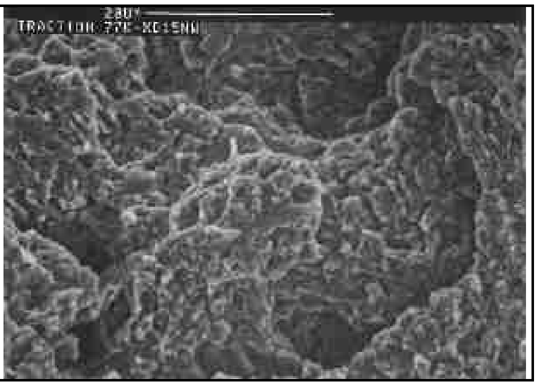


Figure 4 : Current density results

Figure 5 : Fracture surface for XD15N steel

Mechanical properties

The mechanical characterisation program was carried out at room temperature and cryogenic temperature as well (tensile test on 4 samples at room temperature, 77 K and 30 K , hardness, toughness).

The results are given in table 3.

Steel	Test Temp. °C	UTS MPa	Yield 0.2 MPa	Elongation %	K_{1C} MPa√m	Hardness HRc
XD15NW	293	2330	1860	3 .2	17	59
	77	2565	2480	0.7	8.5	
	20	2605	/	/	/	
X105CrMo17	293	1980	1910	1	22	59
	77	2230	/	/	15	
	20	2005	/	/	15	

Table 3 : mechanical properties of materials

Except toughness, the XD15NW steel shows better properties than X105CrMo17. The ultimate tensile stress and yield stress increase when temperature decreases, while toughness decreases. Furthermore XD15NW steel maintains a non zero elongation at 77K.

An " elastic – brittle" behaviour is observed on the surface fracture with a globally brittle intergranular mechanism and a ductile one at the microscopic scale (cups resulting from decohesions between micro-metric precipitates and the matrix - figure 5)

Alternating tension fatigue

Alternating tension tests were carried out in liquid nitrogen using a stress ratio R = σ min / σ max = 0.1. The XD15NW steel held a longer number of cycles than reference steel (figure 6). Large eutectic carbides are responsible of crack initiation for the X105CrMo17, whereas for the XD15NW steel no particular defect initiation could be observed.

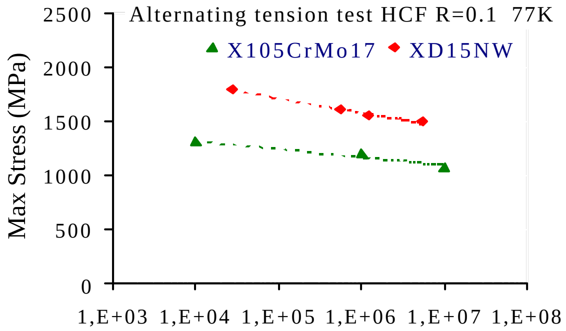


Figure 6 : alternating tension tests in liquid nitrogen

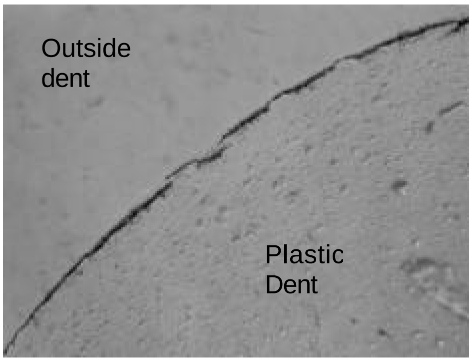


Figure 7 : surface crack on the edge of a dent

Ball on plane Hertzian contact

This test was developed to measure the properties of materials when submitted to high compressive stresses. The load could be either statically or cyclically applied with a 50 Hz frequency. Test were performed in liquid nitrogen with loading conditions corresponding to maximum Hertzian stresses (theoritical elastic calculation) between 4000 MPa and 15000 MPa and a constant 1000 MPa amplitude for fatigue tests.
The crack initiation was detected using acoustic emission technique (piezo-electric sensor 500Hz used to transform mechanical signal into electric signal) that could be used at low temperature.

After testing the plan sample shows a plastic deformation (indent) resulting from the ball loading, that was sized using a 3D profilometer. Consequently, the stabilized Hertzian stresses is lowered.
The plastic strain limit (ratio hd / Dball = 0.0001 with hd = indent depth and Dball = ball diameter) is larger for XD15NW (stabilized Hertzian stress = 4,700 MPa) than for X105CrMo17 (4,600 MPa).
For higher loading, cracks observed using optical analysis initiate most frequently on the annular edge of the plastic strained zone (figure 7), but also inside the dent or in periphery of dent.
The limit for crack initiation after 10^5 cycles is also better for the nitrogen steel.

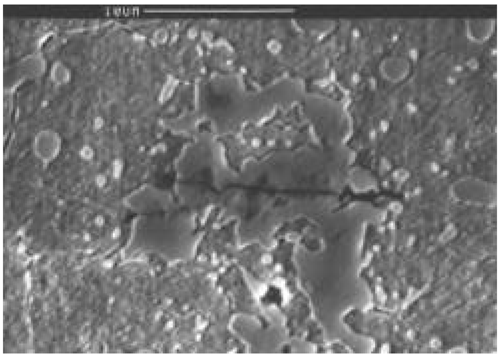


Figure 8 : broken carbide on X105CrMo17

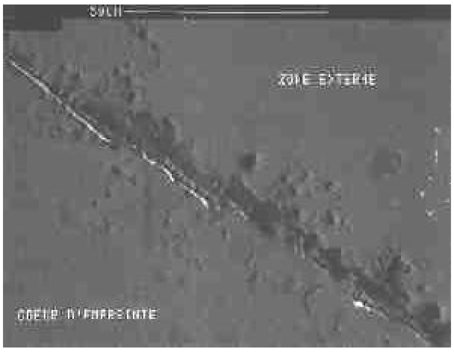


Figure 9 : cracks on XD15NW

Initiation points could be easily identified for X105CrMo17 on large broken M7C3 eutectic carbides located on the edge of the plastic strained zone (figure 8). For the nitrogen steel cracks do not initiate on heterogeneities such as non metallic inclusions or carbides but micro-cracks seem to initiate along the grain boundaries (figure 9). For both materials the crack growth is inter-granular and for the XD15N steel no deep crack propagation was observed.

Rolling contact fatigue

Fatigue life under EHD lubrication conditions was assessed using the SNR FB2 rolling contact fatigue test bench (flat washer test – ball / plane contact).
The test carried out at high Hertzian stress (4.2 GPa) under well controlled EHD lubricating and loading conditions gives the intrinsic fatigue life of the material.
The comparison of the results in figure 10 clearly indicates the fatigue life of low temperature tempered XD15NW is equivalent to those of the high temperature tempered one. Furthermore, the fatigue life of XD15NW is better than the fatigue life of X105CrMo17 steel tempered in equivalent conditions.

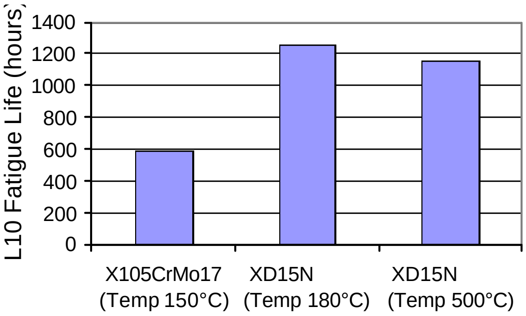


Figure10 : FB2 rolling contact fatigue life results

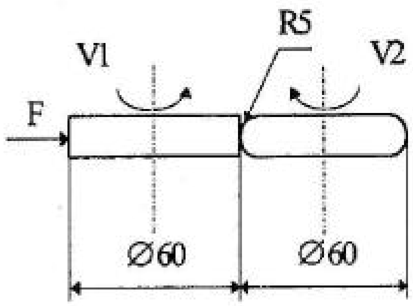
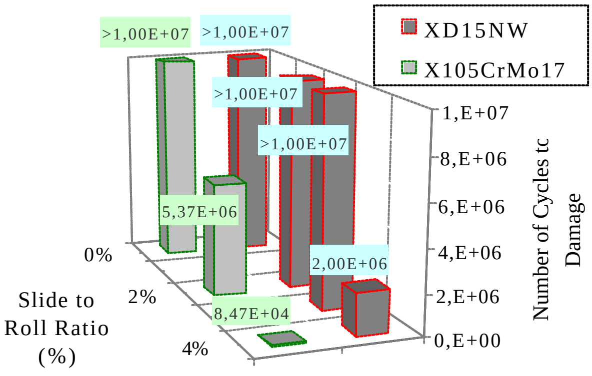


Figure 11 : schematic of the two roller machine

The tribological performance was measured using a high speed two roller cryo-tribometer that makes it possible to run tests under rolling plus sliding conditions, at high speed (10,000 – 10,500 rpm), in liquid nitrogen medium. This machine reproduces the operating conditions at the raceway / rolling element contact of high speed rolling bearings of cryogenic turbopumps.
As shown in figure 11, the tests have been carried out on 60 mm diameter disks that could be driven independently.
Tests were performed, including a 900 seconds running-in stage, for different slide to roll ratio (0 - 1 - 2 - 3 - 4 %) to determine the performance up to the catastrophic increase of the coefficient of friction (tangential / normal load ratio) or suspension at 10^7 cycles. The results are shown in figure 12 .



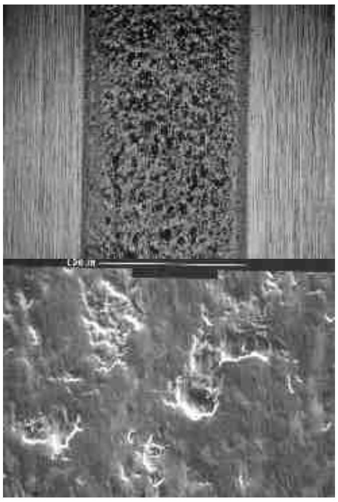


Figure 12 : performance of materials

Figure 13 : wear and surface distress

Whatever the material is, the coefficient of friction increase when sliding is added.
Under rolling plus sliding conditions, fatigue life is reduced and for the same slide to roll ratio, the life of XD15NW steel is longer than those of X105CrMo17.

Surface observations show that for disks stopped before 10^7 cycles, a severe damage occurs (micro-scuffing and skidding). The suspended disks show only a very light wear and a few micro-spalls developed along the rolling direction (figure 13).

Conclusions

The research and development program performed to improve the performances of the liquid hydrogen turbopump bearings of the cryogenic Vulcain engine made it possible to develop and to characterise a new stainless nitrogen martensitic steel.
The metallurgical and mechanical parameters that govern the damaging phenomena of the X105CrMo17 (440C) steel generally used for this application were identified. The limits of this material (corrosion resistance, wear resistance and rolling contact fatigue life) are due mainly to its microstructure containing large carbides and the difficulty to reach high hardness.
The partial replacement of carbon by nitrogen in a medium or high carbon steel leads to a more homogeneous structure with finer eutectic carbides and the addition of elements such as Mo and V harden in a more significant way.
It follows that martensitic nitrogen steel XD15NW™, manufactured using a classical steelmaking process shows an improved corrosion resistance, a good rolling contact fatigue life owing to the hardness that could be achieve after heat treatment and better cryogenic properties.
The fine micro-structure free from large carbides contributes also to explain the significant improvements observed for the structural fatigue characteristics (pulsating axial tension) and the contact fatigue resistance (monotonic and pulsating).
Furthermore, the fatigue life under rolling plus sliding conditions in cryogenic medium is also improved.
Finally, the results obtained justify the use of this steel for the manufacturing of cryotechnic bearings for aerospace applications where requirements in term of reliability are of primary importance and
For which no crack initiation can be tolerated.

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