

Article

Advanced Heat Treatments and Complex Ferritic Structures for Bearing Steels

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Abstract: Nanostructured bainitic steels exhibit an optimum strength/toughness combination as a consequence of their extremely fine structure. They have also demonstrated potential for wear-resistance applications. The aim of this work was to develop bearing steels by the multi-scale control of complex ferritic structures, designed using atomic transformation theory and processed by novel heat treatments. Based on the results, the new ball bearings outperformed conventional grades, approaching more expensive material options.

Keywords: steel; bainite; ultra-high strength; wear resistance; ball bearings

1. In-Use Properties of Advanced Bainitic Steels

Bainitic microstructures might offer an inexpensive solution for application to bearings, based on their interesting combination of fatigue and wear resistance [1]. Considerable progress has been made since the early bainitic steels. The addition of Si, in particular, has allowed for the suppression of cementite, giving an ultra-fine microstructure, consisting of an intricate mixture of bainitic ferrite and austenite. High silicon bainitic steels, usually referred to as carbide-free bainitic steels, attain excellent strength–toughness combinations (up to 1700 MPa and 130 MPa·m^{1/2}), comparable to considerably more expensive high Ni maraging steels [2,3], and have higher wear resistance than pearlitic and conventional tempered martensitic steels [4–7].

Carbide-free bainitic steels derive their strength essentially from their ultra-fine grain size, with typical plate thickness being below 1 µm. Recent advances in the understanding of the bainitic transformation have been exploited to refine bainitic structures to the nano-scale by austempering at low temperatures (125–350 °C) and through careful alloy design [8]. The further grain refinement allowed some to achieve unforeseen combinations of strength and toughness (up to 2500 MPa and 40 MPa·m^{1/2}) in economical steel grades [9–11].

This low-temperature bainite reaction results in a nano-scale ferritic structure with a hardness that has never been achieved before in bainitic steels (>700 HV). Much of the strength and hardness of

the structure comes from the extremely thin platelets of bainitic ferrite, but also dislocation forests and solution strengthening. It is known from X-ray diffraction (XRD) analyses and confirmed by atom probe tomography (APT) that bainitic ferrite contains much more carbon in solid solution than it is consistent with paraequilibrium thermodynamics conditions [12–15].

The different strength-ductility combinations observed in nanostructured bainite are associated with the stress and strain-induced martensitic transformation of retained austenite during tensile testing [9,11,16–20]. Additionally, the formation of twins in austenite films has been identified as a strain hardening mechanism contributing to ductility in these structures [20].

Complex properties, such as wear resistance and fatigue endurance, must be also assessed before the commercialization of novel bainitic steels. Earlier work [4,5] showed that the wear resistance of carbide-free bainitic steels was comparable to that of the best pearlitic grades. In the context of rail applications, Chang [6] investigated the rolling/sliding wear performance of several high silicon bainitic steels, along with detailed microstructural characterization. Results showed lower wear rates in carbide-free bainitic steels and a beneficial effect of the austenite embedded in the sub-micron ferritic structure. In fact, Zhang et al. [21] found that high silicon bainitic steels show better wear resistance than much harder martensite. Likewise, Yang et al. [22] observed that material performance improved when the transformation temperature decreased and a nano-scale bainitic structure was formed.

These studies hint at the considerable benefits of nanostructured bainite against several wear and surface damage mechanisms [7,23,24]. Retained austenite is considered to be critical for improving wear resistance in these structures, since this phase provides hardening by transformation into martensite [23,25]. It is well-known that hardness affects the stress needed to deform the material in the rolling/sliding contact, and it is an important factor in decreasing material loss. Wang et al. [26] found that the austenite in the vicinity of a sliding surface decomposes under the influence of high shear strains, resulting in the formation of an extremely fine structure with grains of ferrite only about 3 nm in size.

Rolling contact fatigue (RCF) has also been determined to be a key mechanism of material removal in the wear performance of nanostructured bainite under rolling/sliding conditions [23,27]. Adhesive forces at the surface are crucial in generating crack initiation sites for RCF cracks. The microstructure was shown to lineate along the sliding direction, establishing preferential paths for cracks to grow along.

The RCF performance of nanostructured bainitic steels, of paramount importance in the context of bearings, has rarely been studied. Liu et al. [28] suggested that nanostructured bainite could prolong the RCF life by effectively retarding crack initiation and propagation in a microstructure composed of nanostructured bainite, martensite, and undissolved primary carbides, with a hardness on par with commercial bearing steels (650–800 HV). However, the specific role of the different constituents was not addressed, and the structures observed were not uniform. Later work by Solano-Alvarez et al. [29] showed that the RCF damage in nanostructured bainitic steels is quite different from that observed in conventional bearing steels. In particular, the formation of voids was prominent at interfaces between regions of bainitic ferrite and martensite, originating from the strain-induced transformation of the austenite blocks. Although void formation was identified as the key mechanism of damage evolution, cracks formed by the linking of voids generally exhibit considerable branching, delaying final fracture.

Further work is required to assess the failure mechanisms and to compare the RCF lives of advanced bainitic steels against conventional bearing steels. In this work, bearing components have been manufactured with a new steel grade, specially designed to transform to nanostructured bainite containing certain amount of austenite, and tested in comparison to reference grade 100Cr6 under severe rolling conditions, such as artificial pollution. Based on industrial trials, it was found that nanostructured bainitic grades offer a competitive solution in the application of bearings.

2. The Design and Manufacturing of Advanced Bainitic Steels for Bearing Components

A bearing is composed of inner and outer steel rings and rotating balls separated by a cage. The materials from the rings and balls are subjected to cyclic loading and unloading, leading to rolling

contact fatigue. In case of good lubrication conditions, the cyclic shear stresses are mainly located in the subsurface. A crack can initiate, on a defect acting as a stress raiser, often non-metallic inclusions. When the crack propagates to the surface, a spalling occurs and leads to the bearing failure, as illustrated in Figure 1. In case of severe operating conditions (deficient lubrication, contaminated lubrication, etc.), shear stresses also occur close to the surface and may lead to a surface-initiated crack, pitting, and spalling.

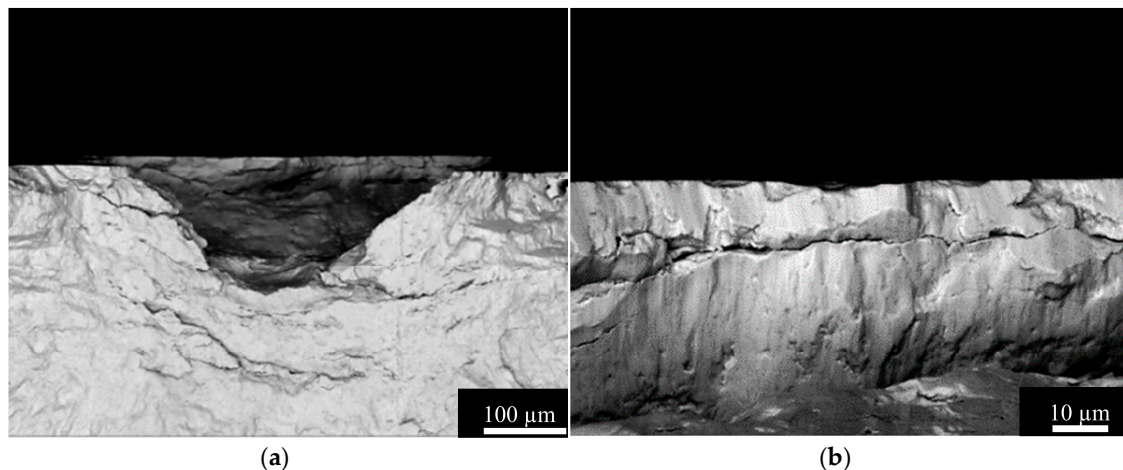


Figure 1. Example of SEM images of cross section of surface fracture on a bearing racetrack: (a) general view and (b) detail of sub-surface cracks [30].

The typical specifications of bearing steels required to resist rolling contact fatigue, are listed in Table 1 based on the properties of 100Cr6 steel after quenching and tempering at 170 °C, a commercial material commonly used in this application. Moreover, cleanliness and thermal stability are vital in bearing steels due to the strict requirements in terms of fatigue resistance.

Table 1. Typical specifications for bearing steels.

Properties	Target Value
Hardness	650–800 HV
Yield Strength	>1600 MPa
UTS	>2200 MPa
Fracture Toughness	>20 MPa√m
Hardenability	Higher than that of 100Cr6 and comparable to that of 100CrMo7

Comparing the properties of nanostructured bainitic steels recently reviewed elsewhere [31] with the industrial specifications of commercial bearing steels (Table 1), a new alloy was designed applying displacive transformation fundamentals to ensure low bainite reaction temperatures by using a high carbon content (1.0 wt.%), the prevention of cementite precipitation from austenite by high silicon additions (1.25 wt.%), and finally, improving hardenability and limited transformation times by an optimum combination of manganese and chromium additions, as Table 2 shows, together with the former alloying developments.

The new material was manufactured as bars after an industrial assessment of feasibility and ensuring high cleanliness for bearing applications. Bars were spheroidized with the exception of 38 mm diameter bars, which were delivered for prototype manufacturing. The hardness of the as-received material was measured to be 230 HV30.

Table 2. Chemical composition of nano-scale bainitic steels, wt.% [8].

Steel	C	Si	Mn	Cr	Mo	V	Nb	Co	Al
First Generation									
Nanobain 1	0.79	1.59	1.94	1.33	0.30	0.11	-	-	-
Nanobain 2	0.83	1.57	1.98	1.02	0.24	-	-	1.54	-
Nanobain 3	0.78	1.49	1.95	0.97	0.24	-	-	1.60	0.99
Second Generation									
Nanobain 4	0.99	1.58	0.76	0.45	-	-	-	-	-
Nanobain 5	1.00	1.53	0.75	0.51	-	-	0.02	-	-
Nanobain 6	1.01	1.51	0.82	0.46	0.10	-	-	-	-
Nanobain 7	0.98	2.90	0.77	0.45	-	-	-	-	-
Nanobain 8	0.88	1.54	0.69	0.50	-	-	-	-	-
Nanobain 9	0.67	1.60	1.25	1.50	-	-	-	-	-
Nanobain 10	0.61	1.45	0.76	2.42	-	-	-	-	-
Nanobain 11	0.64	1.60	1.27	1.50	-	-	0.03	-	-
Nanobain 12	0.58	1.63	1.29	1.43	0.10	-	-	-	-
Nanobain bearing steel	0.98	1.27	0.99	1.36	0.02	-	-	-	0.04

It is well known that this new generation of steels exhibits relatively slow transformation kinetics during bainite reaction [8,32]. Among all the alloying elements, carbon has the strongest effect of retarding bainite formation. In this sense, low austenitization temperatures without full solution of the spheroidized carbides were used prior to bainite transformation. This way, the amount of carbon in the austenite matrix is lower than the nominal, leading to heat treatment times more realistically applicable at an industrial scale. Therefore, different austenitising and bainite transformation conditions were investigated by dilatometry, as Table 3 illustrates.

Table 3. Austenitising and austempering conditions investigated, and the corresponding transformation times, hardness values, and retained austenite contents of the resulting microstructures, as determined by dilatometry.

Austenitising	Austempering	Transformation Time (h)	Hardness (HV30)	Austenite Content (%)
840 °C/30 min	220 °C/16 h	9.3	714	8.3
860 °C/20 min	220 °C/24 h	8.8	710	10.6
	250 °C/15 h	4.3	650	11.5
	270 °C/6 h	2.3	617	12.5
900 °C/30 min	220 °C/48 h	17.9	672	16.1

For that purpose, a Bahr DIL 805D high-resolution dilatometer (TA Instruments, New Castle, DE, USA) and cylindrical samples 4 mm in diameter and 10 mm in length were used. During dilatometry measurements, the specimen is held between two quartz rods, one fixed and the other connected to a linear variable displacement transducer (LVDT). Thus, during austempering heat treatment, it was possible to monitor bainite reaction by measuring the relative change in length of the sample. The time to end bainite transformation is identified at the time at which the dilatation signal reaches a steady-state, and transformation does not proceed any further.

Transformation time values in Table 3 suggest that an increase in the austenitising temperature results in higher transformation times and lower final hardness for the same transformation temperature. Nevertheless, higher austenitising temperatures lead to higher fractions of retained austenite in the final microstructure, due to a higher carbon content in the parent austenite. On the other hand, comparing results obtained at 220, 250, and 270 °C for an austenitising temperature of 860 °C, the primary importance of transformation temperature for the resulting hardness is clearly shown.

After this preliminary study, three different treatments, austempering, quenching and partitioning (Q&P), and quenching and tempering (Q&T), with identical austenitizing conditions (900 °C for 30 min) were applied to obtain bainitic and martensitic structures containing a substantial amount of retained austenite in the designed steel. Figure 2 schematizes the applied heat treatment routes. Bainitic structures were obtained by direct quenching to an isothermal transformation temperature between B_s and M_s after austenitization. On the other hand, Q&P and Q&T martensitic structures were obtained by quenching at a temperature between the M_s and the M_f , 70 °C and room temperature (RT), respectively, followed by a reheating up to a specific partitioning or tempering temperature. Note that a conventional Q&T process requires quenching at RT, but does not necessarily imply quenching at a temperature above the M_f ; though it is the case for the studied steel that the M_f is below RT. Three different temperatures (230, 280, and 350 °C) were selected for transforming to bainite, and for reheating Q&P and Q&T martensitic structures. The transformation temperatures and holding times are summarized in Table 4.

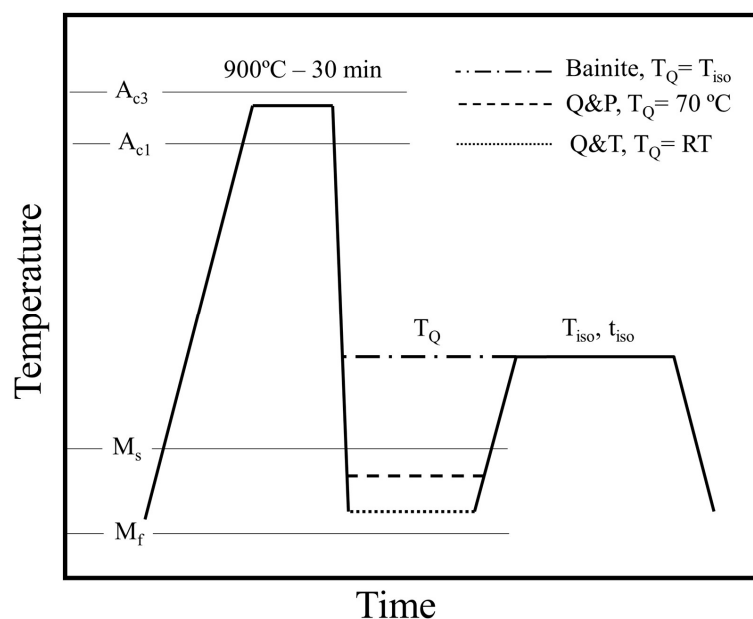


Figure 2. Scheme of the heat treatments performed to obtain bainitic, quenching and partitioning (Q&P), and quenching and tempering (Q&T) martensitic structures in the bearing steel designed. T_Q is the quench temperature, T_{iso} and t_{iso} are the isothermal heat-treatment temperature and time, respectively, and M_s and M_f are the martensite start and finish temperatures, respectively.

Table 4. Heat-treatments performed in Nanobain bearing steel. T_Q is the quench temperature, and T_{iso} and t_{iso} are the isothermal heat-treatment temperature and time, respectively.

Sample	Austenitising	T_Q , °C	T_{iso} , °C	t_{iso} , h
B-230	900 °C/30 min	230	230	35
B-280	900 °C/30 min	280	280	15
B-350	900 °C/30 min	350	350	4.5
QP-230	900 °C/30 min	70	230	100
QP-280	900 °C/30 min	70	280	3.6
QP-350	900 °C/30 min	70	350	2.0
QT-230	900 °C/30 min	RT	230	80
QT-280	900 °C/30 min	RT	280	2.0
QT-350	900 °C/30 min	RT	350	2.0

The Q&P heat-treatment was designed so as to obtain a substantial volume fraction of retained austenite (~50%) before partitioning. According to calculations, the retained austenite content obtained

after quenching to RT of Nanobain bearing steel is ~20%. During tempering, it is known that areas of retained austenite could undergo bainite transformation in a manner similar to that observed during austempering. However, these areas were not evident, due to their relatively small fraction. The purpose of interrupted quenching, T_Q , at 70 °C, was thus to increase this fraction.

3. Complex Ferritic Structures for Bearing Applications

The complexity of nanostructured bainite has been recently reviewed elsewhere [31,33,34] showing the intricate phase distribution and carbon accumulation at the nearly atomic scale. The rationalization to their unprecedented mechanical performances was possible only after the comprehensive examination of the structure at several length scales using a wide variety of techniques, including scanning electron microscopy (SEM), X-ray diffraction analysis (XRD), atom-probe tomography (APT), and electron backscatter diffraction (EBSD).

Following the same procedure, SEM observation in this work was performed on a field emission gun (FEG)-SEM HITACHI S-4800 (Hitachi, Ltd. Tokyo, Japan), operating at 7 kV or 10 kV. Observations were made in the cross section of dilatometry samples. Moreover, EBSD orientation maps were acquired by means of a FEG-SEM ZEISS Auriga Compact (ZEISS, Oberkochen, Germany) equipped with an Oxford NordlysNano EBSD detector and assisted by the AZtecHKL acquisition and OXFORD HKL CHANNEL 5 analysis software (Oxford Instruments, Witney, UK), operating at 15 kV. Analyses were performed in the longitudinal section of dilatometric samples. A $60 \times 45 \mu\text{m}^2$ area was scanned for all samples with a step size of 0.07 μm . Body-centered cubic (bcc) ferrite, face-centered cubic (fcc) austenite and orthorhombic cementite phases were successfully indexed. Finally, XRD measurements were carried out with a Bruker AXS D8 diffractometer (Bruker, Billerica, MA, USA) equipped with a Co X-ray tube, Goebel mirror optics and a LynxEye linear position sensitive detector. XRD data were collected over a 2θ range of 35 to 135° with a step size of 0.015°.

Figure 3 shows FEG-SEM micrographs for bainitic, Q&P, and Q&T martensitic structures in the designed steel, isothermally heat-treated at 230, 280, and 350 °C. Bainitic structures, shown in Figure 3a–c, consist of bainitic ferrite, retained austenite, and undissolved primary iron carbides. Here, the amount of retained austenite increases with increasing the transformation temperature, as Figure 4 shows. Moreover, microstructures obtained after the Q&P (Figure 3d–f) and Q&T (Figure 3g–i) heat-treatments consist of tempered martensite, bainitic ferrite, retained austenite, and undissolved primary iron carbides. In these Q&P and Q&T routes, the steel is quenched at a selected temperature to produce a controlled fraction of austenite. During the following isothermal treatment, carbon migrates from martensite to austenite to increase the austenite stability, resulting in higher austenite fractions at RT after cooling (Figure 4). For both Q&P and Q&T routes, FEG-SEM micrographs do not reveal clear differences in the microstructures obtained for the three different isothermal temperatures. Anyhow, it is expected that the amount of bainite obtained in Q&T microstructures is lower than that in the Q&P structures, since the amount of austenite after the first quenching is also lower (Figure 4).

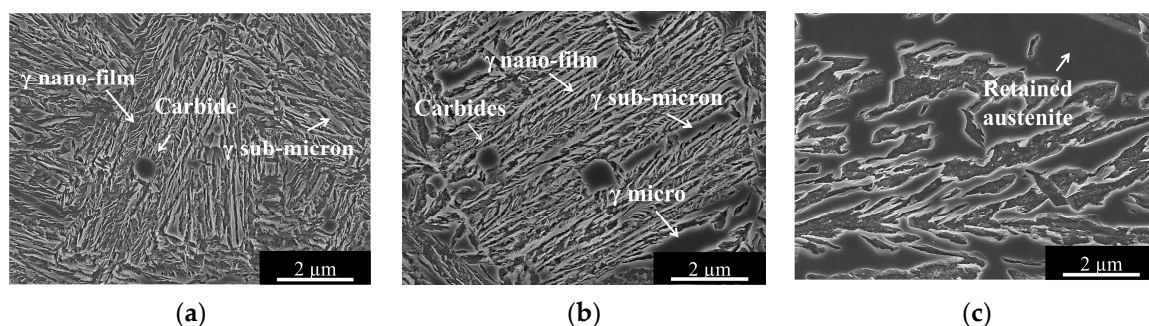


Figure 3. Cont.

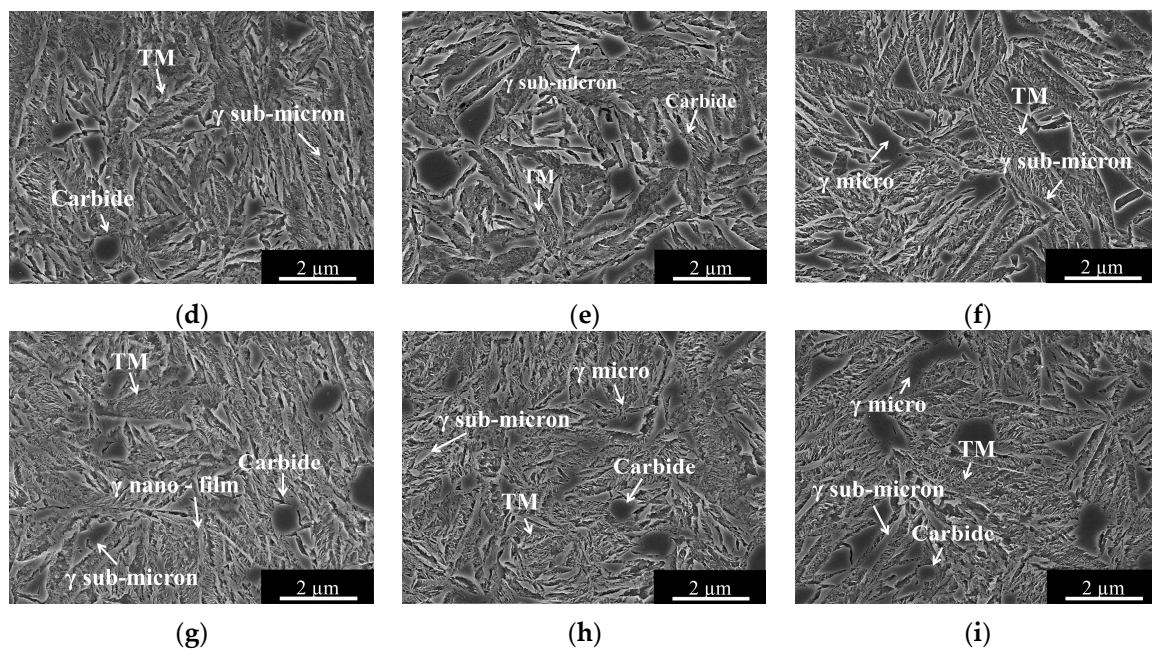


Figure 3. Field emission gun (FEG)-SEM micrographs for samples austenitized at 900 °C for 30 min and isothermally transformed in Nanobain bearing steel at (a) 230, (b) 280, and (c) 350 °C; Q&P samples isothermally treated at (d) 230, (e) 280, and (f) 350 °C; and Q&T samples isothermally treated at (g) 230, (h) 280, and (i) 350 °C. Coarse carbides are undissolved primary iron carbides; TM denotes tempered martensite.

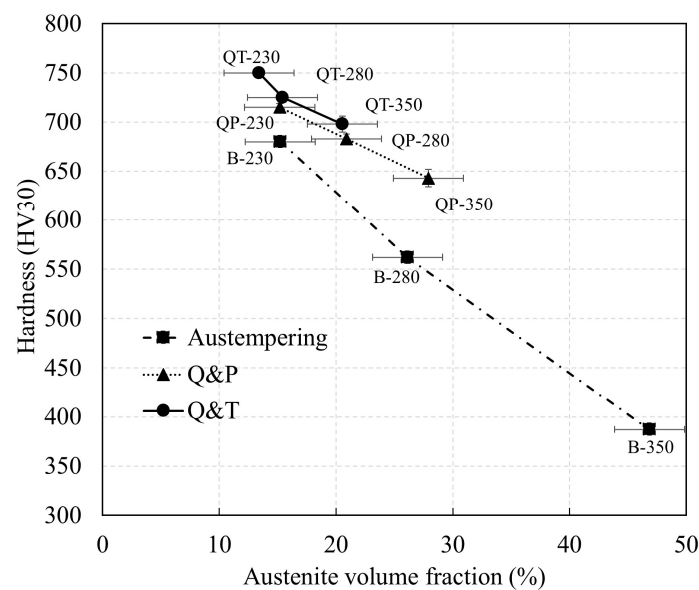


Figure 4. Hardness measurements of bainitic and Q&P and Q&T martensitic structures in Nanobain bearing steel as a function of the retained austenite volume fraction, as measured by XRD.

As shown in Figure 4, bainitic structures reveal far greater sensitivity to the isothermal temperature used in the second step of the heat-treatment than martensitic structures. Indeed, the measured retained austenite content varies from 15% to over 40% when increasing the transformation temperature from 230 to 350 °C, while for Q&P and Q&T structures, retained austenite content remains in the vicinity of 13%–28%. Moreover, with the temperatures in use, it was not possible to achieve the 700 HV required for RCF applications using austempering heat-treatment.

Low-magnification EBSD phase maps for the bainitic, Q&P, and Q&T martensitic structures formed at 230 °C in Nanobain bearing steel are shown in Figure 5. As the inverse-pole figure (IPF) color map images illustrate, nanostructured bainite, Q&P, and Q&T martensitic structures exhibit the same hierarchical ferritic sub-structure of blocks and packets. Blocks are aggregates of plates of ferrite of an equal variant, or slightly misoriented variants, and packets are adjacent blocks with a $\{1\ 1\ 0\}$ ferrite plane almost parallel to the same $\{1\ 1\ 1\}$ austenite plane [35,36]. In consequence, the microstructure obtained was highly misoriented, featuring various potential microstructural barriers. These crystallographic barriers are the boundaries of prior austenite grains, packets, blocks, and ferrite/austenite interfaces.

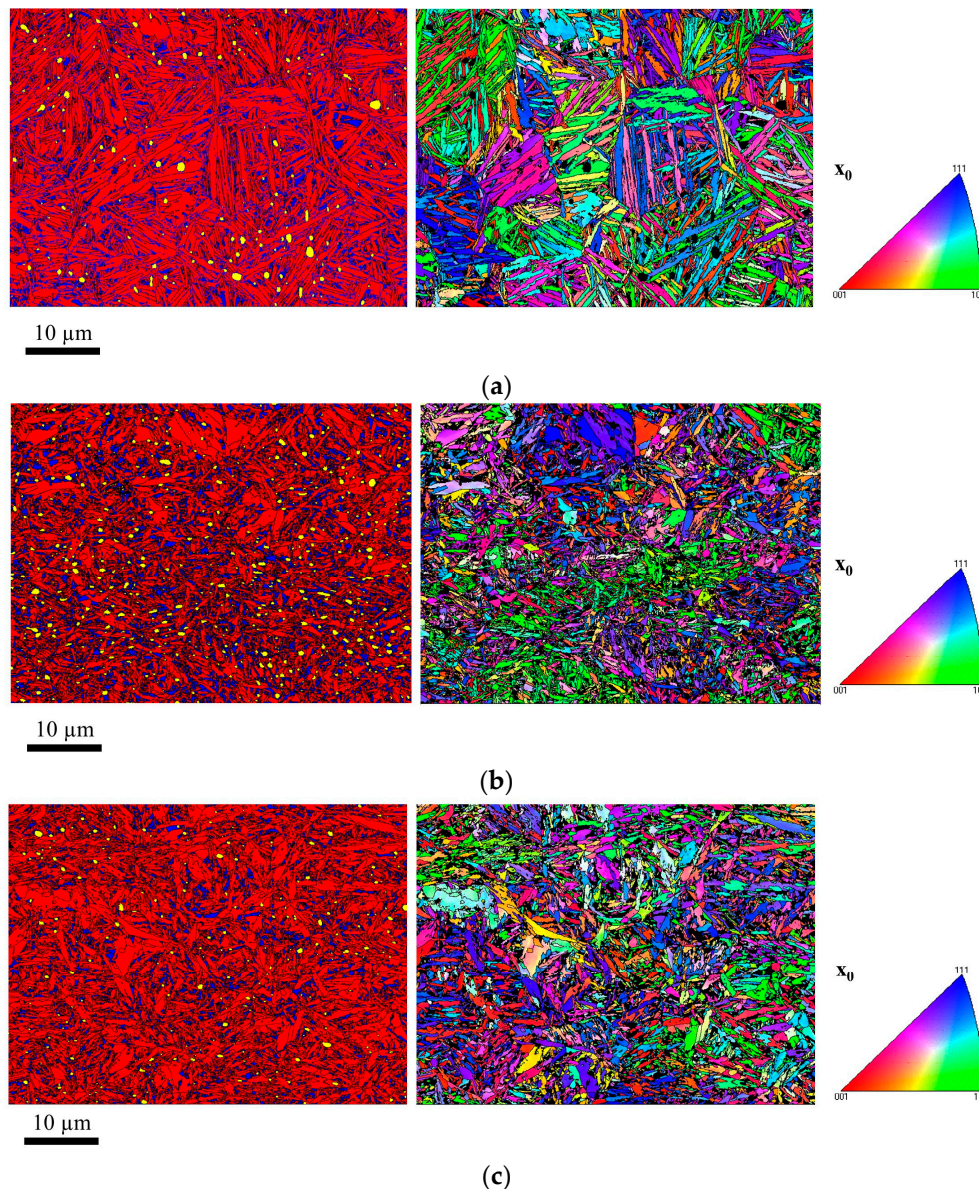


Figure 5. Electron backscatter diffraction (EBSD) phase maps with body-centered cubic (bcc) ferrite in red, face-centered cubic (fcc) ferrite in blue, and undissolved primary carbides in yellow; and corresponding bcc inverse-pole figure (IPF) maps for (a) bainitic structures, (b) Q&P, and (c) Q&T martensitic structures treated at 230 °C.

In previous work [37], a microstructural examination at the fatigue crack propagation showed that a growing crack deflects at the interphase boundaries between blocks and packets of the ferritic

phase, but not in the interphase boundaries within a single block. This suggests that the bainite block size is the crystallographic parameter controlling the crack propagation. A simple visual inspection of the IPF maps in Figure 5 indicates that the bainitic microstructure could exhibit lower resistance to crack propagation during fracture because of its larger crystallographic block size.

4. Mechanical Properties of Selected Ferritic Structures for Industrial Investigations

Heat treatments in Table 5 were selected to be performed on tensile and toughness samples (impact and fracture toughness) with the aim of obtaining bainitic and martensitic structures with different amounts of retained austenite, ranging from 10%–15%, and hardness values in the range of 650–800 HV, as required for bearing applications (see specifications in Table 1). Based on kinetics results in Table 3, two different austenitization conditions (860 °C/20 min and 900 °C/30 min) were investigated. Hardness values and preliminary tensile and toughness (impact and fracture toughness) results in Table 5 confirmed that the resultant microstructures are mechanically interesting.

Table 5. Heat treatments conditions selected for industrial investigations and preliminary mechanical properties.

Austenitising	Treatment	Hardness (HV30)	Austenite Content (%)	YS (MPa)	UTS (MPa)	Charpy (J/cm ²)	K _{IC} (MPa $\sqrt{m}$)
860 °C/20 min	Austemp. at 200 °C/58 h	740	10.1	-	-	6.2 ± 0.3	-
	Austemp. at 220 °C/24 h	710	10.6	1979 ± 21	2292 ± 29	5.4 ± 0.9	-
	Austemp. at 250 °C/15 h	650	11.5	-	-	-	25 ± 0.05
	Q&T at 230 °C/2 h	750	10.2	-	-	3.4 ± 0.2	-
900 °C/30 min	Austemp. at 220 °C/48 h	672	16.1	-	-	-	-
	Q&T at 280 °C/2 h	738	15.4	1824 ± 9	2191 ± 23	-	-

4.1. Rolling/Sliding Wear

Cylindrical rings with Ø45 mm outer diameter, Ø25 mm inner diameter, and 10 mm thickness were machined from a cylindrical steel bar. Three samples were treated in a salt bath following an austempering heat treatment at 220 °C for both austenitization conditions (860 °C for 20 min and 900 °C for 30 min), and a Q&T treatment at 280 °C for 2 h after austenitization at 900 °C for 30 min. As Table 5 shows, they result in bainitic and martensitic structures with different initial hardness values, and in general, adequate yield strength (YS) and ultimate tensile strength (UTS) for bearing applications (see specifications in Table 1). Afterwards, they were ground, in order to remove superficial decarburized and oxidized layers, to the approximate thickness of 0.2 mm.

The rolling/sliding wear tests were carried-out using a UTM 2000 twin-disc machine under unlubricated conditions and in ambient atmosphere. The ring samples were mounted on a two servo drives which could be controlled independently in terms of speed and rotational direction. A load of 300 N was applied to self-mated rings rotating with a 5% slip ratio. The angular speeds of 100 and 95 rpm were tested. The samples were ultrasonically cleaned in an acetone bath, in order to remove loose wear debris and allow for the observation of the wear track morphology. The weight loss of samples was measured using a digital weighing balance with a precision of 0.01 mg. The specific wear rate (SWR) was calculated by the equation $SWR = \text{volume loss (mm}^3\text{)}/\text{Load (N)} \times \text{sliding distance (m)}$ using the sum of volume loss of the two rings [23,27]. The worn surface was analyzed after wear test in order to identify the active wear mechanisms using a JEOL JSM-IT300LV scanning electron microscope (JEOL Ltd., Tokyo, Japan).

The coefficient of friction and SWR results from rolling/sliding tests are plotted in Figure 6. As shown, the coefficient of friction is not influenced by microstructure and worn-surface hardness. For all the investigated materials, the overall average steady-state friction coefficient lies on ~0.6. In contrast, SWR was found to decrease with worn-surface hardness. Beside the differences on the volume fraction of undissolved cementite for different austenitizing temperatures (5 ± 3 for 860 °C for 20 min and 3 ± 3 for 900 °C for 30 min) and mean sizes ($0.6 \pm 0.2 \mu\text{m}$ for 860 °C for 20 min and

$0.8 \pm 0.2 \mu\text{m}$ for 900°C for 30 min), the lowest SWR was achieved in the bainitic sample formed at 220°C after austenitization at 900°C for 30 min with the highest austenite content (16.1%), suggesting that the strain hardening that implies the transformation-induced plasticity of the austenite can influence the rolling/sliding wear behavior of nanostructured bainitic steels.

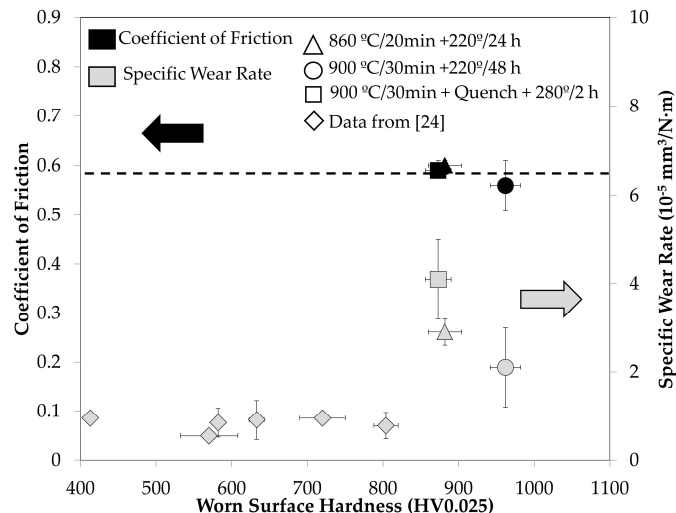


Figure 6. Coefficient of friction (black points) and specific wear rate (grey points) as a function of the worn surface hardness of bainitic structures formed at 220°C after austenitization at 860°C for 20 min (circles) and 900°C for 30 min (triangles); martensitic structures formed after austenitization at 900°C for 30 min, quenching, and tempering at 280°C for 2 h (squares) in Nanobain bearing steel; and bainitic and tempered martensitic structures obtained in a 1C-2.3Si, as reported elsewhere [24].

Rolling/sliding results indicate that wear resistance of nano-scale bainitic structures outperform tempered martensite in the same steel. The three studied structures of Nanobain bearing steel showed equivalent performance to former Nanobain grades [23,24,27]. Even though it was not possible to improve upon Nanobain performance, it is noteworthy that the new material combined the same performance with improved hardness. Similarly, an unexpected lack of correlation of wear resistance with initial hardness was formerly observed on dry-sliding wear of carbide-free bainitic steels elsewhere [38–40], and more recently on nanostructured bainite and tempered martensite of high carbon high silicon steels after dry reciprocating-sliding wear tests conducted at low frequencies against an alumina ball [24]. It was suggested that the highly-refined microstructure and the strain-induced transformation of austenite films increases further surface layer hardening and resistance to material removal by plastic deformation.

The average surface roughness (R_a) was measured before and after wear test detecting an increase from $0.18 \mu\text{m}$ to $\sim 1.2 \mu\text{m}$ for all the tested samples. The worn surface was characterised by an irregular pattern with flakes and minor material build-up as the main features. This can be caused by adhesive wear together with oxidation of produced wear debris during adhesion. Moreover, the nature of rolling/sliding motion contributes to the formation of this type of surface features [41]. Adhesive and oxidative wear mechanisms were identified in rolling/sliding wear after SEM examination of the worn surfaces as shown in Figure 7 for the bainitic structure formed at 220°C after austenitization at 900°C for 30 min.

During running-in and steady-state periods, surface asperities undergo elastic-plastic deformation and oxidation. Therefore, materials can be removed in the form of flakes and wear particles due to oxidation and plastic deformation. The size of the wear debris was found to vary from nano to micro-scale. Likewise, EDS revealed that wear debris contains a significant amount of oxygen ($\sim 43 \text{ wt.}\%$) implying that the oxidation takes place during the wear process (Figure 7d). These oxidized wear debris can be accumulated, agglomerated, and then form oxidized patches (Figure 7c,e,f). Further

fragmentation of oxidized wear debris can cause indentation and plastic deformation when it comes to tribological contact [23,42].

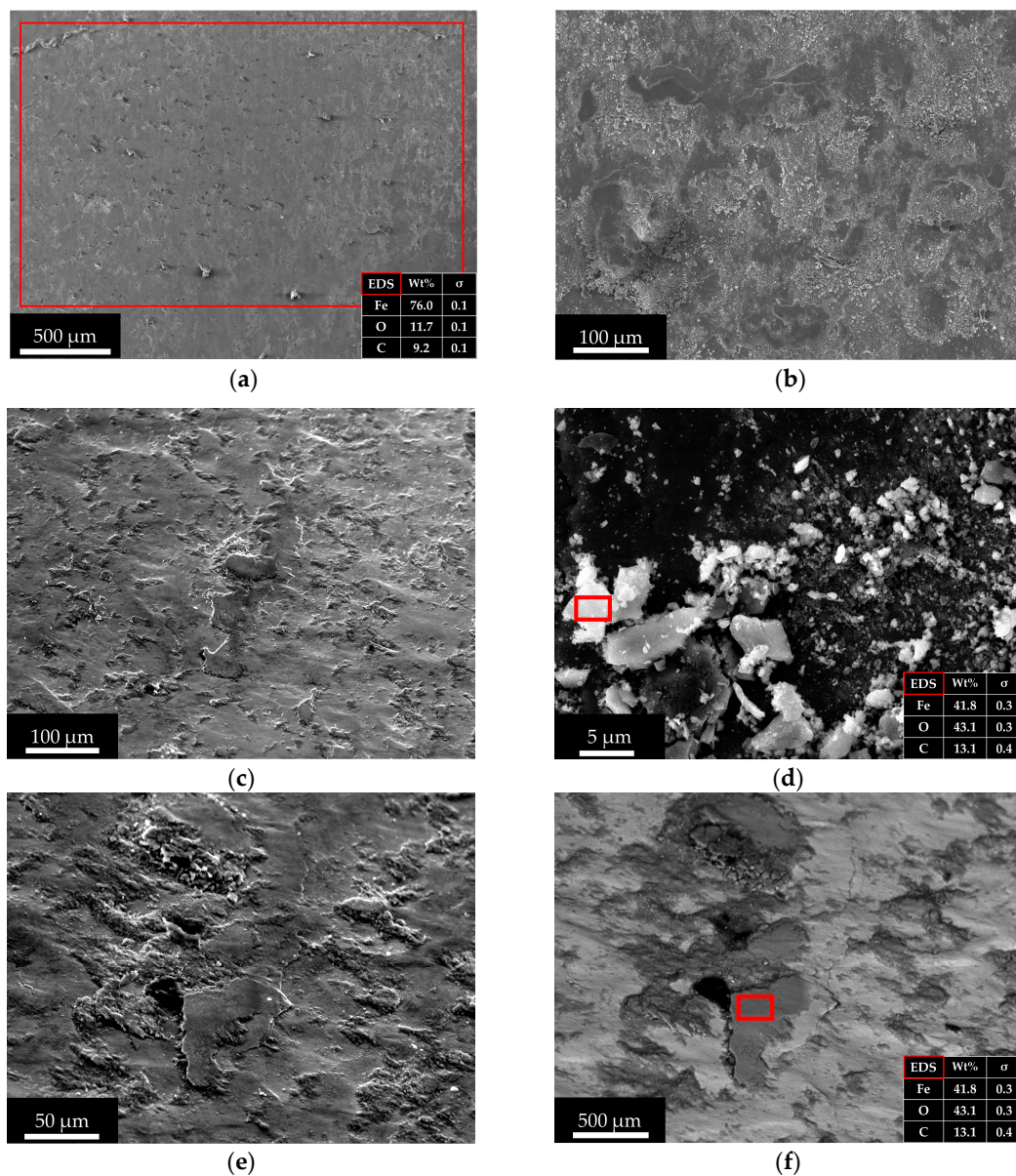


Figure 7. (a), (b), (c), (d) and (e) secondary scanning electron microscopy (SE-SEM) micrographs and (f) Backscattered scanning electron microscopy (BS-SEM) micrograph of the worn surfaces and wear debris after rolling/sliding wear of the sample transformed to bainite at 220 °C after austenitization at 900 °C for 30 min. Red squares represent the EDS analysis areas.

4.2. Contact Fatigue

In order to evaluate the contact fatigue behavior in the same three samples, monotonic and cyclic loads were applied with a spherically tipped conical diamond indenter. Micro Combi (CSM Instrument, Needham, MA, USA) was used following the experimental parameters described in Table 6. To evaluate the reproducibility of the data, at least three indentations were done in different zones of the tested samples. Load displacement curves were obtained and the maximum indentation depth was plotted against the number of cycles. Residual imprints were examined by FEG-SEM Carl Zeiss SMT Inc (ZEISS, Oberkochen, Germany). Their topography was also characterized using an optical confocal microscope Sensofar Plu 2300 (Sensofar, Barcelona, Spain).

Table 6. Monotonic and cyclic loading conditions.

Monotonic loading conditions:	Rockwell diamond tip of 10 μm in radius Maximum load of 2 N Loading rate of 4 N/min Unloading rate of 4 N/min
Cyclic loading conditions:	Rockwell diamond tip of 10 μm in radius Maximum load of 2 N Minimum load of 0.02 N 400 cycles Loading rate of 4 N/min Unloading rate of 4 N/min

The performance of the different materials in cyclic loading conditions was evaluated by analyzing the penetration depths during the indentation test. The curves of the maximum penetration depth as a function of the number of cycles are plotted in Figure 8 for bainitic structures formed at 220 °C after two different austenitization conditions (860 °C for 20 min and 900 °C for 30 min) and martensitic structures formed after austenitization at 900 °C for 30 min, quenching, and tempering at 280 °C for 2 h in Nanobain bearing steel. Results are compared with the contact fatigue performance of the commercial bainitic 100Cr6 steel. Changes in penetration depth are related to hardening (decrease in penetration depth) or softening or damage accumulation (increase in penetration depth). Once again, nano-scale bainite austenitized at the lower temperature (860 °C) in Nanobain bearing steel showed the best repeatability in the response during the test.

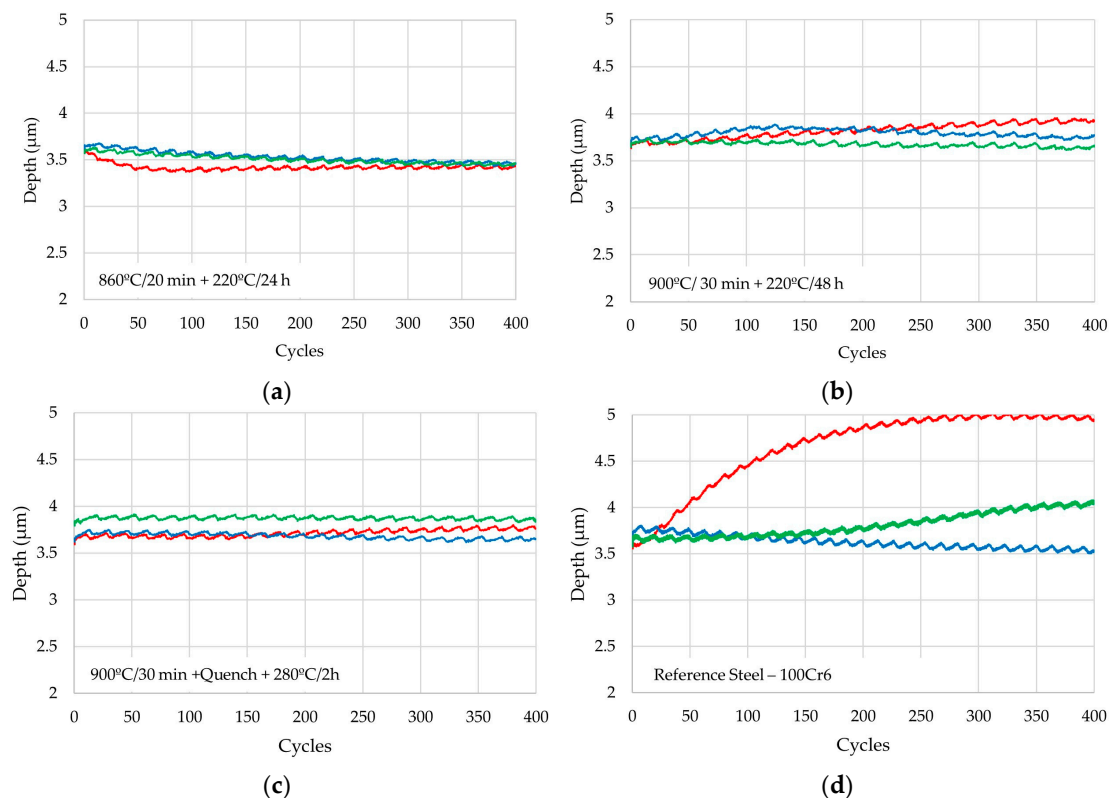


Figure 8. Contact fatigue performance, evaluated in terms of maximum penetration depth as a function of number of cycles. Nanobain bearing steel transformed to bainite at 220 °C after (a) austenitization at 860 °C for 20 min (b) and 900 °C for 30 min; and to (c) tempered martensite after austenitization at 900 °C for 30 min, quenching, and tempering at 280 °C for 2 h, in comparison to (d) commercial 100Cr6 bainitic steel. For each case, three indentations were done.

Furthermore, the comparison of the residual penetration depths in the monotonic and the cyclic indentations in Figure 8 shows that nano-scale bainite formed at 220 °C after austenitization at the lowest temperature (860 °C for 20 min) presents the lowest increment in depth (5%) after applying a maximum load of 2 N during 400 cycles. This means that these structures are less sensitive to contact fatigue than other commercial bainitic steels, such as 100Cr6, which exhibits an increment in depth of 10%.

Examination of residual damage showed large affected areas around the imprints in bainitic samples treated at 220 °C after austenitization at 900 °C for 30 min and in the tempered martensitic samples, even when the penetration depths of residual imprint remained constant. Commercial steels with a high sensitivity to contact fatigue usually reveal evidence of large pile-up of material and broken carbides under cyclic loading conditions [1]. In contrast, the surface of cyclic indented bainitic samples treated at 220 °C after austenitization at 860 °C for 20 min did not present any evidence of damage, which indicates a good performance in contact fatigue conditions.

5. Industrial Trials

Industrial components were manufactured from 38 mm bars of the newly designed Nanobain bearing steel to explore its RCF performance under severe rolling conditions, during transformations to nanostructured bainite and tempered martensite. For that purpose, an industrial heat of 100 tons was produced and approximately 20 tons were hot rolled and processed into material for scaling up the investigation. Based on contact fatigue results in Section 4.2 and hardness values in Table 5, three different heat treatments were selected to be performed on components, with the aim of obtaining bainitic and martensitic structures at the same austenitization temperature (860 °C) and with hardness values higher than 700 HV. Thus, bainitic samples were transformed at 200 and 220 °C for different times (see Table 5) and a martensitic sample was quenched and subsequently tempered at 230 °C for 2 h. The three structures achieved contained approximately 10% of retained austenite (Table 5).

Prototypes were manufactured in the geometry of a ball bearing, as shown schematically in Figure 9. Inner and outer rings were manufactured from Nanobain bearing steel grade. Standard balls and cages were used. The prototype test bearings were first rotated with oil added with contamination in the shape of hard metal particles to create representative damage on the raceways. The bearings were then cleaned and run in clean lubrication using the test conditions presented in Table 7.

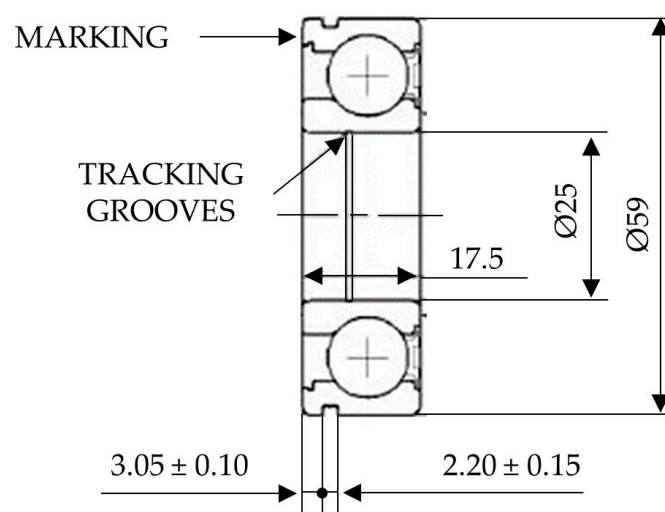


Figure 9. Geometry of the manufactured prototypes.

Component lifetimes were calculated using Adonis Reliability software (QLIO Annecy transfert, Annecy, France) based on the Weibull method. Results are listed in Table 8. Both nano-scale bainitic structures showed similar performance. On the other hand, the best endurance was achieved in the

tempered martensitic structure. This can be partially explained by the higher hardness of the structure (750 HV) resulting in lower damage during natural pollution of the bearings.

Table 7. Rolling contact fatigue (RCF) testing conditions for industrial components.

Magnitudes	Testing Values
Axial load	330 daN
Radial load	660 daN
Rotation speed	2250 rev/min
Hertz pressure	2.8 GPa
Fit on shaft	M5

Table 8. Lifetime L10 calculations performed for components tested in artificial pollution tests.

Treatment	No. of Bearings	Spalling Number	L10 in Hours-90% Confidence Limit			β Slope
			Lower Limit	L10	Higher Limit	
Austemp. at 200 °C/58 h	14	8	75.4	111.7	165.5	2.9
Austemp. at 220 °C/24 h	16	8	55.5	104.1	195.1	1.67
Q&T at 230 °C/2 h	16	8	137.1	233	396	2.01

In addition, tests on flat washer samples with indentations were performed. Four dents were generated on each ring using a Rockwell indenter (AMETEK, Inc., Devon-Berwyn, PA, USA). The rings were then tested in clean lubrication at a maximum Hertzian pressure of 3.5 GPa to ensure failure initiation on dents. If no failure occurred after 400 h, the ring was suspended. Between six and 12 rings were tested after austempering at 220 °C for 24 h and Q&T at 230 °C for 2 h. After testing, the rings were examined to track spall initiation and propagation.

Surface fatigue in a ring typically begins with a crack (spall) that is V-shaped (Figure 10a). The spall grows (Figure 10b) until material flakes away from the surface (Figure 10c). In this case, bainitic components proved to endure the test conditions better, even though crack propagation, once initiated, was faster, according to the Adonis software, than in martensitic washers, as Figure 10 illustrates.

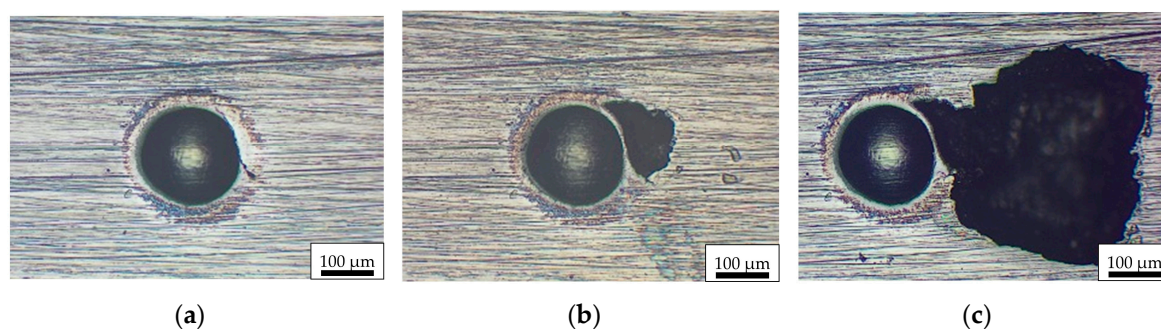


Figure 10. Evolution of spalling on a dent generated on a ring with a nano-scale, bainitic structure formed at 220 °C for 24 h: (a) 36 h, (b) 48 h, and (c) 60 h.

6. Conclusions

Based on microstructure characterization of a new steel grade, specially designed to transform in a reasonable time to nanostructured bainite, and its performance in RCF and rolling/sliding, it was concluded that nanoscale bainitic structures exhibit an optimum combination of contact fatigue and sliding wear performances. This is related to their ultra-fine grain size, ultra-high hardness, limited damage observed related to plasticity, and the homogeneity of these structures.

The new Nanobain steel showed competitive performance in terms of components' lifetimes in the bearings' industrial trials. Nanostructured bainitic components proved to endure the artificial pollution test conditions better.

However, the application of the advanced Q&P and Q&T treatments in this newly developed grade outperformed nanostructured bainite in terms of crack propagation. This is thought to be related to the finer effective crystallographic structure of martensite obtained with this newly developed grade.

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