Surface Characteristics Analysis of Gas Carburized New Hot Working Tool Steel

M. L. Fares,* M. Athmani, and Y. Abidi
Mechanics of Materials and Plant Maintenance Research Laboratory (LR3MI),
Metallurgy and Material Engineering Department,
Bajdi Mokhtar University, BP 12, Annaba 23000, Algeria
(Received 6 October 2011; Accepted 4 November 2011; Published 10 December 2011)

Gaseous carburizing effects on improvement of performance of a new grade hot working tool steel close to as either chromium AISI H11/H13 are investigated. Such treatments are performed at 930°C for various processing times. Formed layers that are characterized by their basic properties (i.e. thickness, depth, formed phases, hardness distributions, carbon/carbides distributions and redistribution of alloys in the carburized layers) show a close dependence of both time-temperature process and chemical composition of material. Test results indicate that retained austenite amount act to improve significantly the wear resistance of components despite their relatively lower hardness. In addition, it is addressed that performance and productivity of the as-new material could be extensively enhanced when the above treatment is used. Metallurgical evaluations are carried out using metallographic techniques, optical, scanning electron microscopy equipped with an energy-dispersive X-Ray spectrometer and X-ray diffraction techniques. Mechanical properties are achieved mainly by standard hardness and wear tests.

[DOI: 10.1380/ejssnt.2011.430]

Keywords: Carburizing; Diffusion and migration; X-ray diffraction; Hardness; Wear resistance; Retained austenite

I. INTRODUCTION

Hot forming operations, like forging dies, aluminum extrusion dies and any other operations producing: trimming dies ejectors and cutting punches, place difficult demands on tool steels. The temperatures involved limit the steels that are suitable for use. Most hot working tool steels require a combination of high-temperature strength (i.e. hot hardness and temper resistance), wear resistance and toughness. In applications involving thermal cycling (intermittent cooling, etc.), heat check resistance is also required. Many standard steel qualities (e.g. AISI H10-H19) and several commercial special ones exist specifically for these applications. Traditionally, H13/H11 are the most common heat treatable tool steels used in this area, while they offer reasonable high temperature strength and toughness, but not high hardness or wear resistance [1]. Friction at the die-billet interface (known as the bearing) and the corrosive environments [2] generates a high amount of wear. The usual practice, acting to attain a successful future of components, is to select the most economical steel grade, appropriate heat treatment and surface hardening sequences, which consistently meet the desired properties.

For instance, conventional heat treatments, (i.e. quenching and tempering) can have a vast impact on steel parts in terms of mechanicals properties; i.e. toughness, shock resistance, dimensional stability, high hardenability and wear resistance [3, 4]. Such treatments, related to specific applications, are expected to lead to hardness levels of 43-52, 44-50 and 40-55 HRC for extrusion dies, die-casting and forging dies respectively [5]). However, because of severe operating conditions, as quoted earlier, these operations turn out to be not sufficient. Then, it becomes necessary to search for other solutions, including surface modifications, which can ensure the sought design, the performance and the durability of the finish product.

Commonly, case hardness can be extensively increased by diffusion of carbon and/or nitrogen into the surface, using thermochemical treatments (i.e. carburising [6-8], carbonitriding [9-11] and nitrocarburizing processes [12, 13]. Among these popular processes, gaseous carburizing is a hardness treatment of controlled carbon diffusion into the surface of a component. It is achieved at temperatures generally over 900°C, followed by quenching and tempering operations, with the main objective of increasing the component’s surface hardness. Solid and liquid carbon-carrying medium may be employed in these processes since gaseous carburizing is extensively applied in industrial scale for mechanical parts. It is well known that many factors such as hardness, case depth, residual stress, surface finish, microstructure, grain size, globular and network carbides, intergranular oxidation as well as the presence of retained austenite can affect the usefulness of carburized steel. Hardening is accomplished when the high-carbon surface layer is quenched to form martensite, so that a high-carbon martensitic case with good wear and fatigue resistance is superimposed on a tough steel core [14]. However, if the material is quenched directly after austenitizing, retained austenite will form along with a course grain size and a high heat distortion will occur. The risk of cracking is reduced if the carburized steel is quenched in oil, depending on the steel alloy and its component shape.

One of the important factors of carburized steels is the effective case depth, which is defined as the distance below the surface when the hardness is equal to 550HV [6]. Case depths are a function of carburizing temperature-time and the available carbon potential at the surface while case hardness is primarily a function of the carbon content. The time necessary for making deeper case depths can be reduced by high temperatures related to the process [15]. When prolonged carburizing times are used for deep case depths, a high carbon potential produces a high surface-carbon content, which may thus result in excessive formation of carbide networks and retained austenite. These two microstructural elements may have adverse effects on the distribution of residual stresses in the case-hardened part. Consequently, a high carbon potential may be suit-
able for short carburizing times but not for prolonged ones. During the treatment, the characteristic future of the formed microstructure is the appearance of important carbide networks, which can produce brittle cases, leading to tooth end chipping [16]. Besides, it has been reported that carbides precipitation does not unfavorably influence properties [17]. In another hand, it is well known that excessive retained austenite may cause soft cases and lowers surface hardness and should be avoided since its effect on mechanicals properties is still controversial. Some investigators state that a relatively amounts of retained austenite, in the case of carburised components, is beneficial to contact fatigue resistance [18], while others believe that retained austenite should be minimized [19]. Another particularity of this process may be the internal oxidation [20]. This latter is relative to the presence of some alloying elements and their quantities in solid solution within the steel while its extent is a function of temperature-time of carburizing processes.

Although, a large number of published works are focusing on carburizing of standard steels, few researches related to carburizing process on hot working tool steels exist in the literature. For instance, a single study has been conducted by Babul et al. [21] on the pack carburizing process at 930°C of both GOST 4H5W2FS and ASM 6437E steel grades (close to AISI H13 and H11 tool steels, respectively). Hence, this investigative work aims at demonstrating the usefulness of industrial new hot working tool steels (comprising higher carbon content and lower silicon content), which may have effect to enhance performance of the components. Experimental data, consisting in metallurgical evaluations of relevant microstructural parameters, from closely monitored gaseous carburized treatment has been added to the available published data in order to increase search providing a good scientific foundation. It is hoped that this overview will lead to obtain competitive materials having better tool life and higher productivity. Detailed phase characterizations in terms of chemical composition, formed phases and microstructures are undertaken mainly by optical microscopy (OM) and scanning electron microscopy (SEM) as well as X-ray diffraction techniques (XRD). Mechanical properties, comprising hardness and wear resistance, are evaluated by microhardness and abrasive standards tests, respectively.

II. EXPERIMENTAL PROCEDURES

For the present investigation, modified alloy hot working tool steel (provided by SA. Euro Metal Service (Schildé, Belgium) was considered. Compared to AISI H11 and AISI H13 steels, it discloses some difference in carbon, vanadium, nickel and silicon contents. Chemical composition, identified by the Thermo Scientific ARL 4460 optical emission spectrometer (OES), is listed in Table I.

Gaseous carburizing treatments were carried out in a gas atmosphere at heat treatment industrial plant (ENMTP-CPG-Constantine, Algeria) using treatment cycles and treatment parameters, which are presented in Fig. 1 and Table II, respectively. The purpose of the suggested cycle is to achieve both satisfactory case depth and improved properties (e.g. hardenability; wear resistance and toughness). In the first step, cubic samples with size of about 10×10×10 mm³ were subjected to the annealing treatment in order to eliminate residual stresses that resulted from machining operations. Then, carburized specimens carried out at temperature of up to 930°C, were quenched in oil bath, from 830 to 180°C and finally tempered at 200°C for 2 h. For analysis, surface preparation consisted of a mechanical grinding using 250, 400, 800, and 1000 grit silicon carbide papers and subsequently polishing down to a final step of 1 μm diamond pastes. After an ultrasonic cleaning in ethanol, samples were etched in a 3% nital solution at ambient temperature. Detailed metallographic investigations to reveal structures, constituting the basic morphology and the extent of thickness surfaces of the treated samples, were achieved using optical microscope type GX.51 Olympus and a scanning electron microscope JEOL-6500F type. XRD patterns were acquired using a Philips PW1730/10 diffractometer, operating at 40 kV and 200 mA with a cobalt anticathode of λCo = 0.179026 nm. A Dosophatex sample holder and an Elphyse position sensitive detector were also used. A 2θ scan was recorded within the range 35 to 105° with a constant step wise increase of 0.04°/15 s. Both retained austenite and carbide contents were assessed by the Guinier’s method [22], which is described in the Appendix. At this stage, to reduce the cutting strain effect, induced by the mechanical polishing.

TABLE I: Chemical composition of studied steel (wt %), Fe balance.

<table>
<thead>
<tr>
<th>Designation</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Al</th>
<th>Cr</th>
<th>V</th>
<th>Ti</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-received steel</td>
<td>0.52</td>
<td>0.42</td>
<td>0.55</td>
<td>0.22</td>
<td>0.03</td>
<td>5.20</td>
<td>0.34</td>
<td>0.004</td>
<td>1.10</td>
</tr>
<tr>
<td>Standard 1.2343/H11</td>
<td>0.38</td>
<td>0.40</td>
<td>1.00</td>
<td>-</td>
<td>-</td>
<td>5.10</td>
<td>0.40</td>
<td>-</td>
<td>1.25</td>
</tr>
<tr>
<td>Standard 1.2344/H13</td>
<td>0.40</td>
<td>0.40</td>
<td>1.05</td>
<td>-</td>
<td>-</td>
<td>5.15</td>
<td>1.00</td>
<td>-</td>
<td>1.35</td>
</tr>
</tbody>
</table>
TABLE II: Industrial gaseous carburizing parameters and specimen designations.

<table>
<thead>
<tr>
<th>Process treatment</th>
<th>Treatment parameters</th>
<th>Exposure time (h)</th>
<th>Sample designations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Endothermic carrier gas:</td>
<td>(39.36% H₂, 0.23% CO₂, 19.83% CO, 0.76% H₂O) that is enriched by methane. Carbon potential: 0.8</td>
<td>1</td>
<td>C1</td>
</tr>
<tr>
<td>Gaseous carburising</td>
<td></td>
<td>2</td>
<td>C2</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>C3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4</td>
<td>C4</td>
</tr>
</tbody>
</table>

FIG. 2: Optical micrographs of gas carburized specimens at selected holding times (a) 1 h, (b) 2 h, (c) 3 h, and (d) 4 h.

III. RESULTS

Figure 2 shows typical cross-sections of case hardened microstructures of the carburized specimens as a function of exposure times of 1, 2, 3, and 4 h. In all associated samples C1, C2, C3 and C4, a respective fine oxide layer is obvious on outer surface. It corresponds to the soot that is one particularity of the gaseous carburizing process. However, oxides are not revealed by X-ray diffractograms in Fig. 3 since they are removed by means of electropolishing techniques to ensure accuracy measurements of retained austenite content. Figure 4 exhibits SEM microstructures of the sample C2 (after 2 h of exposure) for different magnifications and positions, as indicated in the optical micrograph of Fig. 2(b). It is worthily noticeable that internal oxidations are not present on grain boundaries. However, it is likely to have more internal oxidation for higher durations, such as obvious in Fig. 2(d) for C4 sample.

Table III summarizes the distribution of both retained austenite and martensite contents of each carburized specimen C1, C2, C3 and C4, assessed from X-ray diffraction results, as depicted in Fig. 3. Austenite due to its structural difference from other phases in steel produces a diffraction peak at different location than ferrene.

TABLE III: Assessments of martensite and retained austenite contents for selected carburized specimens obtained by means of X-ray diffraction data (Gunier’s method [22]).

<table>
<thead>
<tr>
<th>Sample designations</th>
<th>Fe α (%)</th>
<th>Fe γ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1</td>
<td>71</td>
<td>29</td>
</tr>
<tr>
<td>C2</td>
<td>79</td>
<td>21</td>
</tr>
<tr>
<td>C3</td>
<td>95</td>
<td>5</td>
</tr>
<tr>
<td>C4</td>
<td>97</td>
<td>3</td>
</tr>
</tbody>
</table>
FIG. 4: SEM micrographs of sample C2 at different magnifications and locations according to the optical micrograph of Fig. 2(b). (a) and (b) location 1, (c) location 2, and (d) location 3.

rite/martensite. Accordingly, the microstructure of the sample C1 consists mainly of tempered martensite superposed to Fe$_3$C added to a large amount of retained austenite close to 29% that was untransformed into martensite. As carburized time increases, retained austenite content decreases to 21% for sample C2 while some amount of carbide M$_7$C$_3$ is formed (M= Fe, Cr, Mo mainly). In Figs. 4(a) and (b), corresponding to location 1, carbides are precipitated in a network-like fashion along grain boundaries at the surface. Moreover, others finely dispersed carbides are noticeable within grains. Figure 4(c) corresponds to location 2, which is the crossing point between the surface and the core. It is noticeable that carbides are of elongated forms. Finally, in Fig. 4(d), which corresponds to location 3, the core exhibits a microstructure composed of tempered martensite, retained austenite and fine granular carbides (Fe3C). These latter present a strong tendency to change from elongated to spherical shapes compared to those of position 2. These sporadically carbides precipitations are characterized by a size that is above the observed average.

According to the C3 sample, the retained austenite decreases significantly down to 5%. This large fall results in an important transformation of the retained austenite into martensite, from 79 to 95%, and in some carbide. At this stage, a new carbide M$_{23}$C$_6$ type (M=Cr, Cr mainly) is formed, to be added to the first type, which is M$_7$C$_3$. Finally, for the sample C4, associated to a longer carburizing holding time, the retained austenite amount becomes close to 3%, which is a low limit. The carbide M$_7$C$_3$ is suppressed because of the lower degree of carbon supersaturation in matrix and the stabilization of retained austenite. As a result, the precipitation of M$_{23}$C$_6$ carbides, embedded in tempered martensite, will progress.

Effective case depths and hardness measurements of C1, C2, C3 and C4 specimens are assessed using microhardness profiles from Fig. 5 and are recorded in Table IV. The size of the hardness impression is affected by a distance limitation from the edge of the specimen at which reliable microhardness measurements can be made with a diamond pyramid indenter for a 100 g load. This lack will give larger indentations and erroneously low hardness values, which are subsequently ignored because surface is not microscopically plane. In all carburized samples, it is noticed that the shape of microhardness profiles is rather similar, exhibiting a decrease in hardness gradients from the surface towards the core. Hardness gradients reveal a lower hardness at the tooth surface followed by a sharp drop with the maximum at a depth of 57 µm below the surface. Particularly, the profile of C4 sample exhibits a significant laying from 217 to 417 µm, corresponding to microhardness values in the range of (950-877) HV$_0.1$; subsequently, a large drop is noticeable. Carbides precipitations related to C4 sample, strongly contribute to the hardness increase since its hardness gradient between the core and the case is twice higher than that of C2. For this later, the established hardness gradient between surface and core position is the same and equal to 200HV, which is indicative of relevant toughness in the present study. The inspection of the related hardness impressions reveals small, irregular and discontinuous cracks that are not in-
TABLE IV: Comparison results of case depths, hardness and weight loss tests in function of holding time for various carburized specimens. Incertitude’s measurements are respectively ±5, ±5 and ±10⁻⁴.

<table>
<thead>
<tr>
<th>Property</th>
<th>C1</th>
<th>C2</th>
<th>C3</th>
<th>C4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Effective case depths (µm)</td>
<td>269</td>
<td>419</td>
<td>620</td>
<td>780</td>
</tr>
<tr>
<td>Surface hardness (HV₀)</td>
<td>649</td>
<td>680</td>
<td>763</td>
<td>789</td>
</tr>
<tr>
<td>Core hardness (HV₀)</td>
<td>513</td>
<td>523</td>
<td>509</td>
<td>511</td>
</tr>
<tr>
<td>Maximum hardness (HV₀)</td>
<td>741</td>
<td>756</td>
<td>856</td>
<td>965</td>
</tr>
<tr>
<td>Global weight loss (10⁻³ g)</td>
<td>47</td>
<td>47</td>
<td>66</td>
<td>107</td>
</tr>
<tr>
<td>Global wear rate (10⁻⁴ g·m⁻¹)</td>
<td>27.80</td>
<td>27.80</td>
<td>39</td>
<td>63.70</td>
</tr>
</tbody>
</table>

![Graph showing wear behaviour of carburized samples at selected holding time](http://www.ssj.org/ejssnt)

FIG. 6: Wear behaviour of carburized samples at selected holding time.

dicative of a brittle material. Hardness measurements of unhardened material show that all selected samples featured similar core hardness, which is located randomly in the range of (509-523) HV₀.

Wear expressed as weight losses vs. sliding distances is presented in Fig. 6. It can be seen that all curves display the same trend. In the range of sliding distance of 0 to 112 m (zone “a”), all selected samples exhibit the same relatively large weight loss value, which correspond to the existence of oxides in the outermost surface. Such formed oxides, of lower hardness and lower adherence, went away very quickly, as confirmed by the average wear-rate value of 89×10⁻⁴ g·m⁻¹. Within the second field (“b”), corresponding to the range of (112-450) m, the weight-loss values present a relative stationary state corresponding to an average wear-rate of 35×10⁻⁴ g·m⁻¹. In the case of C1 and C2 samples, this state can be ascertained by the presence of higher retained austenite contents, which is likely to preserve certain ductility, resulting in an improvement in terms of friction. For C3 and C4 samples, homogeneously dispersed carbides in the hardened layers act to improve wear resistance, which is corroborated by their corresponding hardness levels, as it can be seen in Fig. 5. Finally, from 450 to 1690 m (“c”), it is observed that all specimens present a relatively large increase in weight loss corresponding to an average wear-rate of 43×10⁻⁴ g·m⁻¹. In the range of 1352 to 1690 m, the wear behaviour of C4 sample is characterized by the highest wear-rate of 64×10⁻⁴ g·m⁻¹, although in theory wear resistance depends mainly on the hardness of the effective case depth of carburized samples. Summarizing it is obvious that samples C1 and C2, exhibit an almost same global wear behavior, which is characterized by a better wear performance than those of samples C3 and C4.

IV. DISCUSSIONS

Based on the results from this investigation, several complex transformations occur in these steel during processing. Many factors may affect the mechanical properties of gaseous carburized steel. Those factors include chemical composition, hardness, residual stress, surface finish, microstructure, grain size, globular and network carbides, intergranular oxidation, microcracking as well as the presence of retained austenite. In view of this complexity, it is reasonable to discard the effects of microcracking, surface finish, and residual stress because it is not possible to find any trend with steel species in this specific study. The main factors that may be taken into account are the influence of hardness effect arising from suggested treatments, in addition to both retained austenite and carbides levels.

According to X-ray diffractograms in Fig. 3 and SEM micrographs of Fig. 4, it is worth interesting to note that internal oxidation is not present on grain boundaries. This feature may be due to our chemical composition. In fact, the high carbon potential acts, in a sensitive way, on the reduction of oxidizing effect, depending on the treatment duration [24]. Besides, the presence of nickel (~0.22%) has the potential to reduce the internal oxidation of Fe-Cr-Mn steel, and to increase the reliability of components operating under high loads, as reported by Parrich [24] and Gliner [25], respectively. An et al. [7] also corroborate reductions of internal oxidation when lower silicon content (~0.55%) is added to the material. In the same context, Lohrmann et al. [26] indicate that the internal oxidation, taking place during the heat-up phase, is limited and is not affected by the duration of the heat-up time within the normal range of 50-90 min, which is close to our conditions, particularly for both samples C1 and C2.

During heat treatment of steels with a higher content of carbon and alloy elements, which shift Ms and namely Mf temperatures towards lower values, residual austenite and specific carbides may form in the structure. These specific carbides and their morphologies depend on the alloy composition as well as the processing time used to achieve the
produced hardness at the surface is 790 HV
0

0

position 2). It is well known that the presence of Fe3C carbides, enriched by alloying elements such as chromium, tungsten molybdenum and vanadium, can promote enhancement of wear resistance, as well as static strength of steel components [21]. The carbide formation is promoted by the tempering temperature close to 200°C. As it has been reported, carburized steels tempered close to this temperature exhibit excellent mechanical properties [27]. In sample C3, the extensive transformation of the retained austenite, allows in both associated farther amount of martensite (Fe3C) and precipitation of some alloyed carbides. In this condition, because of the prolongation of the holding time, compared to the one of C2 sample, unfavorable effects may occur, consisting, for instance, in decarburization phenomena. In fact, the carbon potential at the surface may be over 0.8% and the increase of holding time acts to diminish this potential toward the sublayers. Accordingly, as already reported above, it is well known that the decrease of carbon content provokes the increase of both Ms and Mf (martensite start and finish temperatures), resulting in a significant increase of martensite content and in a reduction of retained austenite content, when specimens undergo quenching treatment. In addition, specifically at high temperature, the decrease of carbon potential have the potential to destabilize austenite during quenching which would support the precipitation of carbides in accordance with chemical composition of as-studied steel. With regard to Fig. 3 and Table III, these carbides consist in a newly formed M23C6 carbide type (M=Cr, mainly), added to the one of M7C3 (M=Fe, mainly), which agree with literature data [28].

The presence of higher carbon and carbide-forming additions induces growth of strength and hardenes of martensite, as well as the volume share of retained austenite beneath the surface. The noticed decrease in hardnes gradients, related to samples C1, C2, C3 and C4 (Fig. 5), is owed to a change in relative carbon concentrations. It is well known that carbon diffusivity is relatively lower than in other steel because it contains many carbide-forming elements (Cr, Mo and V). In addition, the presence of silicon, in spite of its lower content, also reduces the degree of carbon diffusivity, resulting in a comparatively thin concentrated carburized layer [29]. The lower hardness depicted at the tooth surface may be due to the presence of oxides and to the edge effect, since there is no support at the edge during the test. The higher microhardness value of 950 HV0.1 of C4 sample, compared to those of C1, C2 and C3 samples, can be ascribed to the retained austenite content, which is less important. Indeed, retained austenite is softer than martensite. A similar core hardness for selected samples, which is located randomly in the range of (509-523) HV0.1, suggests that there is no carburized time effect on the substrate hardnes. For instance, according to results provided by the literature [21], ASM 6437E steel grade, close to AISI H11 type, presents pack carburizing treatment duration of 6 h. The equivalent produced hardness at the surface is 790 HV0.05 with an effective case depth of 400 μm. In our case, the maximum hardness value for a cycle duration of only 2 h (sample C2) is almost similar (756 HV0.1) with a similar effective case depth of 419 μm (Table IV). These results point out an important gain in the percent cycle duration of 71%. This fact can be corroborated by the presence of nickel in solid solution, as claimed by Kozlovskii et al. [30].

Thermochemical treatment makes it possible to create on the working surfaces of steel parts layers with enhanced tribological properties. Carburizing and subsequent quenching-tempering increased the wear resistance. Usually the surface layer is saturated with carbon to a eutectoid or moderately hypereutectoid content. After quenching, the microstructure comprises high-carbon martensite and eventually some amount of carbides (i.e. Fe3C, M7C3 and M23C6). According to our abrasive test results, the effect of retained austenite content is particularly significant in this investigation. In this context, the inquiry of whether the retained austenite phase is beneficial or whether in some cases it can be unfavorable remains ambiguous [31, 32]. Some authors [33] suggest that the transformation of retained austenite (softer phase), to martensite act to increase the yield stress under compression but reduce viscosity, which is favorable for friction coefficient. Others authors stated [10, 24] that the presence of retained austenite, under the effect of the compressive stresses, resulting from abrasion contact, can harden under the sole effect of induced wear hardening (strain accommodation). This state has, as effect, an improvement in terms of friction. In addition, it is also claimed [18] that during abrasive test, the strain accommodation induces ability of retained austenite to evolve into martensite. At the surface vicinity, samples C1 and C2, comprising respectively 29% and 21% of retained austenite content, exhibit a relative slightly better wear resistance compared to C3 and C4 samples, with 5% and 3% of retained austenite content, respectively. On the other hand, for the same treated samples, it is also noticeable that, the decrease of the retained austenite content, from the surface towards the core, results in a relative strong increase of weight losses [6]. Here, the existence and the type of carbides in the martensitic matrix may result in a further increase of abrasive wear by particles.

For C3 and C4 samples, associated wear resistance at the surface “zone b” are almost similar to the ones of C2 and C3 samples. This fact is corroborated by the corresponding higher hardness levels, which is ascertained by Fig. 5. Ever since the relatively large amount of weight losses for C5 sample (“zone d”) can be ascribed to the possible unfavourable carbide because of the higher holding time at high temperature.

Several previously published works dealt with the comparison between steels having a rather higher retained austenite level with very low hardness and rather low retained austenite level with very high hardness. Our experimental results are close to those previously reported [34, 35] and allow stating the achievable improvement of wear behavior in treated surface under the effect of both controversial mechanisms, as above mentioned. Summarizing, performed tests showed that a low surface hardness does not automatically mean that the protection against wear could be reduced [33]. In fact, reliable assessments of a material depend strongly on the induced wear.
mechanism since carbides could likely be detrimental because of their brittleness effect [36].

V. CONCLUSIONS

1. The obtained microstructures from the gas carburizing process, mainly consisted of Fe₃C, M₇C₃ (M: Cr and Mo mainly) and M₂₃C₆ (M: Cr and Mo, mainly) carbides embedded in tempered martensite which might cause an enhancement of a wear resistance as well as a static strength of the steel components.

2. The relatively higher carbon content of the studied material led to the formation of further retained austenite amount, which is maximal for the lower holding times. The tested specimens, with higher retained austenite content, exhibited better global wear resistance in spite of their corresponding lower average hardness.

3. According to these results, in conjunction with those of literature, the presence of lower silicon as well as nickel content in the as-received steel, resulted in a decrease of the internal oxidation of material during the prescribed treatment, which is a consistent result.

4. The recorded treatment time of 2 h allowed obtaining very satisfactory mechanical and tribological properties of carburized parts (i.e. effective case depth-0.419 mm, hardenability-toughness: surface hardness-756 HV₀.1 / core hardness-523 HV₀.1). Such a shorter time was up to 71% less than the obtained according to literature data.

5. Summarizing, the option of gaseous carburizing process on this new hot working tool steel featured very favorable functional properties and the reduction of the total processing time, which acts to minimize overall cycle cost and to enhance component productivity.

Acknowledgments

The authors are grateful to the technical staff of Ecole des Mines de Saint-Etienne, Centre SMS France, for their support and for making available the facilities for various characterization tests.

Appendix: Assessment of percentage contents of ferrite, carbides and retained austenite after heat treatment (Guinier’s Method [22])

\[ I = I_0 \cdot \frac{e^4}{m^2e^3} \cdot \frac{(1 + \cos^2 2\theta)}{2} \cdot \frac{1}{16\pi \sin^2 \theta \cos \theta} \cdot \frac{1}{\chi^2} \cdot \frac{F_{hkl}^2 \cdot n}{V_c} \cdot D \cdot dV, \]  \[ (1) \]

where:
- \( I \): diffracted power on the unit length of a Debye-Sherrer ray recorded at a distance \( r \) from the sample,
- \( I_0 \): intensity per unit area of the incident intensity,
- \( \frac{e^4}{m^2e^3} \): constant = 7.9 \times 10^{-26},
- \( \frac{(1 + \cos^2 2\theta)}{2} \): polarization factor,
- \( \frac{1}{16\pi \sin^2 \theta \cos \theta} \): Lorentz-polarization factor,
- \( \lambda \): wavelength of used radiation,
- \( F_{hkl} \): structure factor depending on crystalline structure,
- \( V_c \): volume of unit cell,
- \( n \): planes multiplicity \((hkl)\),
- \( D \): temperature factor,
- \( dV \): volume element of the powder.

Constants throughout the measured spectrum are mentioned as \( K \). From here: \( I = K \cdot i \cdot dV \).

Rays intensity in the mounting reflection: sample of infinite thickness (relative to the depth of penetration of used X-rays) and at plane surface.

Thickness layer \( dx \) at depth \( x \) allows expressing the intensity:

\[ dI = K \cdot i \cdot S \cdot \frac{dx}{\sin \theta} \cdot \exp \left( \frac{2\mu \rho}{\sin \theta} \right), \]  \[ (2) \]

where:
- \( S \): section of the beam X,
- \( \mu \): mass absorption coefficient of the sample,
- \( \rho \): density of the sample.

The total intensity should be:

\[ \int_{x=0}^{x=\infty} dt = K \cdot i \cdot S \cdot \frac{\mu \rho}{2\mu \rho}. \]  \[ (3) \]

For a mixture of phases, phase \( \alpha \) gives peaks:

\[ i_{hkl}^{\alpha} = K^{\alpha} \cdot i_{hkl}^{\alpha} \cdot S \cdot \frac{\mu \rho}{2\mu \rho}. \]  \[ (4) \]

where:
- \( \mu \rho \): linear absorption coefficient of the mixture,
- \( V_{\alpha} \): Volume fraction of the ferrite phase.

For each phase, it is possible to calculate the values of \( i_{hkl}^{\alpha} \), and then the volume fraction of the ferrite phase should be:

\[ V_{\alpha} = \frac{i_{hkl}^{\alpha} \cdot i_{hkl}^{KB} \cdot F \cdot V_{\alpha}}{\sum(i_{hkl}^{\alpha} \cdot i_{hkl}^{KB} \cdot F \cdot V_{\alpha})} \times 100. \]  \[ (5) \]

This is the same for volume fraction of retained austenite and carbides.
(Ohio State University, Columbus 1988).
(1999).
[13] T. Babul, A. Nakonieczny, and J. Senatorski, in Proc. the 20th Int. Conf. on Surface Modification Technologies
No. 37.