A New 1,400 MPa-Class Pre-Hardened Steel Grade for Plastic Molding and Mechanical Engineering Applications

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INTRODUCTION

Plastic injection moulding requires the use of mould inserts and holders made of high strength steel capable of withstanding cyclic high stresses resulting from high injection pressures. During the plastic injection process, the polymer material, made soft by a temperature of about 200-250°C, is injected into a mould and then cooled to take the desired shape. The choice of materials depends on the injected polymer and the nature of the part. High hardness and mechanical properties are required for durability (wear and fatigue resistance) while good machinability, optimal surface finish after polishing, and good thermal conductivity to remove heat dissipated during cooling of the mould parts are equally important.

The high mechanical properties are obtained by a controlled quenching and tempering process, either of the final part or the base metal. Today, the latter approach is a common choice because cost and development times are reduced when mould parts are directly machined from a high quality prehardened steel block compared to subsequent heat treatment of the final tools [1].

While it is nowadays possible to machine hard materials over a wide range of cutting speeds, prehardened grades are generally available for hardness levels ranging from 30 to 40 HRC only. To achieve higher properties, mould makers are increasingly using ASTM A681 H11 or H13 grades, which are machined in the annealed condition and require final heat treatment of the parts to obtain the desired hardness. Those steels were designed many decades ago for hot working processes such as forging, die casting, and hot stamping where they are exposed to severe working conditions with rapid changes in temperature up to 600°C and high mechanical loads [2]. However, the requirements for the use as plastic injection mould components are lower than for hot working, whereas the high alloying content in H11 or H13 impairs the thermal conductivity for those grades [3], which is a key characteristic for plastic moulding applications.

This study focuses on the production of a new grade with reduced contents of Cr, Mo and V, and having up to a thickness of 250 mm a typical hardness in the delivery condition of 44 HRC (1400 MPa UTS) with excellent machinability. Besides plastic moulding applications, the overall good balance between strength and toughness opens the door to the manufacture of many tools, dies, and machine parts used in mechanical engineering applications.

MATERIALS AND EXPERIMENTAL PROCEDURES

The chemical composition of the Cr-Mo-V steels used in this study is shown in Table 1. We investigated 4 different laboratory casts and an industrial heat of the new grade. For the laboratory tests, heat treatments were conducted in a laboratory furnace.
using different time and temperature parameters. For full scale industrial production, the several plates were manufactured in Industeel production facility in Le Creusot, France, from bottom-poured cast ingots made from electric arc furnace with ladle refining and vacuum degassing to provide a clean and homogeneous steel. The ingots were reheated to a temperature of 1,250°C and hot-rolled to 60 – 80 – 130 mm thickness in a four-high (quarto) reversible mill. To achieve the final properties, the plates were reheated to 950°C prior to roll-quenching under water and subsequently tempered at 580°C.

To follow the microstructural evolution during the tempering stage, in-situ High Energy X-Ray diffraction (HEXRD) experiments were conducted at the DESY synchrotron facility in Hamburg, Germany, on Petra III P07B beamline. The samples were heated at 1°C/s and 100°C/s, from room temperature up to three tempering temperatures: 550, 575 and 600°C and maintained at that temperature for 1 h then cooled at 2.5°C/s. The Debye-Scherrer diffraction patterns were collected continuously along the tempering process. More details concerning the HEXRD experiments are provided in [4].

The mechanical properties were measured according to ISO 6892-1 method B and using round samples machined in the longitudinal direction. Charpy impact testing was performed using standard 55 x10x10 mm samples. Three samples for each condition were tested and their average value was taken as the CVN-impact energy value. For hardness testing, Brinell HBW10/3000 or Vickers indenters with different loads have been used depending on the situation. The samples were etched with 2% Nital for metallography observation using Keyence VHX series 7000 optical microscope and a Zeiss GeminiSEM460 Scanning Electron Microscope (SEM). The amount of retained austenite in the industrial sample was measured by X-ray diffraction.

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>S</th>
<th>P</th>
<th>Si</th>
<th>Cr</th>
<th>Ni</th>
<th>Al</th>
<th>Mo</th>
<th>V</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>L1</td>
<td>0.32</td>
<td>0.89</td>
<td>0.0005</td>
<td>0.002</td>
<td>0.14</td>
<td>3.0</td>
<td>0.8</td>
<td>0.047</td>
<td>0.76</td>
<td>0.29</td>
</tr>
<tr>
<td>L2</td>
<td>0.35</td>
<td>0.89</td>
<td>0.0004</td>
<td>0.0018</td>
<td>0.14</td>
<td>2.0</td>
<td>0.8</td>
<td>0.05</td>
<td>0.77</td>
<td>0.29</td>
</tr>
<tr>
<td>L3</td>
<td>0.35</td>
<td>0.89</td>
<td>0.0004</td>
<td>0.0017</td>
<td>0.57</td>
<td>2.0</td>
<td>0.7</td>
<td>0.049</td>
<td>0.77</td>
<td>0.29</td>
</tr>
<tr>
<td>SP450</td>
<td>0.34</td>
<td>0.97</td>
<td>0.0014</td>
<td>0.0065</td>
<td>0.23</td>
<td>2.1</td>
<td>0.7</td>
<td>0.068</td>
<td>0.73</td>
<td>0.22</td>
</tr>
</tbody>
</table>

Table 1: Chemical composition of investigated laboratory casts (L1 to L3) and industrial material (SP450).

RESULTS AND DISCUSSION

Microstructure and Stability of Retained Austenite

In a first approach, the hardness evolution of the laboratory grades was measured before and after tempering. Samples were austenitized at 1100°C to achieve dissolution of the V-rich MC carbides. Two quenching rates have been selected: a fast water quenching to produce hard martensite and a slow cooling rate of 100°C/h to produce auto-tempered martensite or bainite. All samples were subsequently tempered at 575°C during 1 h. The hardness evolution is displayed in Table 2 and the corresponding optical micrographs of the microstructures are given in Figure 1.

After fast quenching, a hardness superior to 600 HV10 is obtained in all grades. This value decreases to approximately 470 HV10 after tempering. In contrast, samples cooled at 100°C/h showed a hardness increase of approximately 30-40 HV10 after tempering. This is an unexpected behavior where tempering results in an increase in hardness rather than the typical softening. These observations indicate a significant difference in the response to tempering between the fast-quenched samples (martensitic structure) and the slowly cooled samples (auto-tempered structure).

<table>
<thead>
<tr>
<th></th>
<th>Full quench</th>
<th>Full quench +Tempered 575°C 1h</th>
<th>Cooling 100°C/h</th>
<th>Cooling 100°C/h +Tempered 575°C 1h</th>
</tr>
</thead>
<tbody>
<tr>
<td>L1</td>
<td>613</td>
<td>471</td>
<td>432</td>
<td>472</td>
</tr>
<tr>
<td>L2</td>
<td>634</td>
<td>468</td>
<td>393</td>
<td>420</td>
</tr>
<tr>
<td>L3</td>
<td>629</td>
<td>471</td>
<td>403</td>
<td>448</td>
</tr>
</tbody>
</table>

Table 2: Hardness evolution (HV10) after quenching or slow cooling followed by tempering at 575°C during 1 h.

The key difference between the fast and slowly cooled specimens is the significant amount of retained austenite obtained after slow cooling. The study of austenite decomposition during the tempering process was performed by in-situ HEXRD for grades...
L2 and L3. Figure 2 depicts the evolution of the austenite mass fraction as a function of temperature and soaking time for different tempering temperatures 550, 575 and 600°C, with a heating rate of 1°C/s and a final cooling rate of 2.5°C/s.

At room temperature, in the as-quenched state, austenite mass fractions of about 12 and 25% are measured for grades L2 and L3 respectively. The latter contains more silicon, well known to promote the stabilization of austenite. The salient feature of the results is that the austenite fraction remains virtually unchanged during heating and soaking. Decomposition occurs mainly during the final cooling. The higher the tempering temperature, the higher the start temperature of austenite decomposition. The gradual shift of austenite decomposition to higher temperature with increasing the tempering temperature from 550 to 600°C can be clearly seen in Figure 2(c). The final austenite mass fractions are about 4 and 10% for grades L2 and L3 respectively.

From these observations, it can be argued that:

(i) Carbon depletion of the retained austenite occurs during the heating and soaking stages of tempering. This can be caused by direct precipitation in the supersaturated austenite or carbon diffusion from supersaturated austenite to the tempered matrix.

(ii) During the final cooling, the carbon-depleted austenite forms fresh and hard martensite in the final microstructure, resulting in an increase in hardness compared to the as-quenched condition.

In most cases, austenite is expected to decompose during heating and holding into ferrite and carbides [5] or lower bainite [6,7]. The absence of decomposition during heating and soaking followed by a transformation into fresh martensite on final cooling is only seldom reported [8,9]. In situ HEXRD method used in this study gives a new and direct evidence of this behavior.

Figure 1: Light Optical micrographs of the specimens for grades: (a) L1 after quenching at 100°C/h; (b) L3 after quenching at 100°C/h; (c) L1 after tempering at 575°C during 1 hour and (d) L3 after tempering at 575°C during 1 hours. LOM microstructural evolution in L2 was found comparable to L1.

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Figure 2: Evolution of the austenite mass fraction during the tempering cycle for grades L2 (0.15% Si) and L3 (0.60% Si) and different tempering temperatures 550, 575 and 600°C (a) heating, with a heating rate of 1°C/s,

(b) soaking for 1 hour and (c) final cooling, with a cooling rate of 2.5°C/s.

Mechanical Properties of Industrial Plates

For industrial production, the challenge is to produce steel plates with good combination of strength and low temperature toughness across the entire thickness section. During the quench process, significant temperature gradients are produced that result in a variety of microstructures, such as martensite, bainite and retained austenite through the thickness of the large section blocks. Avoiding surface cracking of the plate often observed during fast quenching on highly alloyed grades is also of critical importance.

The difficulty is to find a chemical balance. The adequate C content with the addition of substitutional alloying elements to increase the hardenability of the steel even at low cooling rates is necessary to obtain a high hardness. The elements Mn, Mo, V or B play a crucial role in this regard [10]. The elements Cr, Mo and V also promote the formation of fine and stable precipitates and thus have the potential to bring secondary precipitation hardening without degrading other properties. The reduction in chromium content from 5 to less than 3% would also offer a better potential of thermal conductivity but negatively affects the hardenability.

Based on the results of the laboratory study and extensive literature search, the alloying design detailed in Table 1 was proposed for the industrial heat. Figure 3 shows that homogenous hardness of typical 430 – 460 HBW is obtained across the entire thickness of the 60 – 80 and 130 mm, which is higher than the target value of 410 HBW for the new grade and in good agreement with the hardness levels found on the laboratory casts and heat treatments. The tensile and impact properties of the industrial plates are presented in Table 3. All the plates show similar level of strength and elongation. High strength superior to 1400 MPa UTS is obtained, including the mid-thickness (½-t) position. CVN-impact energy of 15-20 J are measured at room temperature, which is relatively satisfactory for this type of grade and thickness.

The microstructure of the industrial material is illustrated in Figure 4 for the 130 mm plate. A lath like structure including abundant inter- and intra-cementite (M3C) precipitation typical of tempered martensite is observed. In Cr-Mo-V steels, the presence of fine alloyed carbides of type M23C6 and MC have the potential to bring secondary hardening during hardening provided their size remains small, typically below 50 nm. It was not possible to confirm their presence with the SEM. Finally, no retained austenite was observed, which is confirmed by XRD measurements giving an austenite mass fraction below the detection limit of 3%.

In summary, the developed steel satisfies a uniform hardness between 430 and 460 HBW, high-tensile properties superior to 1400 MPa UTS and impact properties greater than 15 J in the transverse direction (ST) at 20°C for plates up to 130 mm. A second industrial campaign was launched to extend the thickness up to 250 mm.
Figure 3: Normalized hardness profiles across the thickness for 3 different nominal plate thicknesses: 60, 80 and 130 mm.

<table>
<thead>
<tr>
<th>Plate thickness</th>
<th>Thickness location</th>
<th>YS (MPa)</th>
<th>UTS (MPa)</th>
<th>A%</th>
<th>Z%</th>
<th>CVN-T (J)</th>
<th>CVN-L (J)</th>
</tr>
</thead>
<tbody>
<tr>
<td>60 mm 1/4 t</td>
<td></td>
<td>1281</td>
<td>1438</td>
<td>18</td>
<td>56</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>80 mm 1/4 t</td>
<td></td>
<td>1253</td>
<td>1440</td>
<td>16</td>
<td>50</td>
<td>16</td>
<td>15</td>
</tr>
<tr>
<td>130 mm 1/4 t</td>
<td></td>
<td>1293</td>
<td>1507</td>
<td>14</td>
<td>41</td>
<td>15</td>
<td>17</td>
</tr>
<tr>
<td>60 mm 1/2 t</td>
<td></td>
<td>1260</td>
<td>1446</td>
<td>16</td>
<td>45</td>
<td>12</td>
<td>16</td>
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<tr>
<td>80 mm 1/2 t</td>
<td></td>
<td>1292</td>
<td>1447</td>
<td>15</td>
<td>50</td>
<td>13</td>
<td>11</td>
</tr>
<tr>
<td>130 mm 1/2 t</td>
<td></td>
<td>1215</td>
<td>1420</td>
<td>17</td>
<td>50</td>
<td>14</td>
<td>16</td>
</tr>
</tbody>
</table>

Table 3: Room temperature tensile and CVN-impact properties for 3 different nominal plate thicknesses 60, 80 and 130 mm and 2 different thickness locations 1/4- and 1/2-t. The tensile specimens were machined in the longitudinal direction. Impact test specimens are machined in both longitudinal (L) and transverse (T) directions.

Figure 4: Light Optical (a) and SEM (b) microstructure of the 130 mm industrial plate at 1/4-thickness position.
Other Properties and Applications

Increasing the thermal conductivity of a plastic mould steel leads to significant improvements in productivity, part quality, and cost-effectiveness in plastic injection operations. Faster cooling rates provided by a better thermal conductivity reduce production times and cycles, resulting in improved productivity. They also help to minimize internal stresses within the plastic part, resulting in higher dimensional stability and lower risk of shrinkage. The thermal conductivity was determined from room temperature to 600°C by the indirect method by multiplying the thermal diffusivity, the density, and the specific heat of the material. The measured values are compared to those obtained for a typical H11 grade in Figure 5, showing that the new grade presents better heat transfer properties, especially in the temperature range around 200°C used for plastic injection moulding operations.

![Figure 5: Comparison of thermal conductivity values from room temperature to 600°C between the new grade SP450 and a typical H11 grade.](image)

Mould polishing is an important step in plastic injection: not only it enhances the aesthetic quality of the final product but also plays a crucial role in improving production efficiency by facilitating smooth ejection and preventing sticking issues. A manual polishing test was carried out by a skilled mould steel hand polisher on a sample of surface 75x75 mm using 600 and then 1200 grit paper, followed by polishing with 6 and 1 μm diamond paste. On such a small-scale sample, the new grade is suitable of achieving a mirror-polished finish. An average roughness of Ra = 0.045 μm and a total profile height Rt = 0.250 μm, was obtained, which corresponds to a roughness class N2-N1 according to ISO 1302. This positive result needs to be confirmed in the future on larger parts.

High machinability of mould steel is critical for reducing manufacturing time and cost of the mould, as well as lifetime of the milling tools. Machining of hard material is today possible with the progress in milling tools and techniques, assuming that the material is homogenous in hardness to avoid short distance variations in the cutting forces and heat generation that affect the tool life and surface finish. Machining tests were carried out with the help of Walter Tools from Sandvik group. The appropriate tools and processing conditions for milling, drilling, and tapping were selected based on their extensive database for similar steels grades of the same hardness level. Thanks to its consistent hardness and structural homogeneity, machinability of the new grade was very satisfactory as depicted in Figure 6 and considering that it was possible to exceed the recommended feed rates and cutting speeds without compromising on the quality of the dimensional accuracy, surface finish and chip formation.
Surface treatment, such as gas nitriding or induction hardening, is another key property when applied to plastic mould steel. The hardness increase of the surface layer provides improved resistance to wear, which helps to maintain dimensional accuracy and surface finish of the plastic parts over extended production runs. Where moulds are exposed to corrosive plastics or aggressive moulding conditions, nitriding also improves the corrosion resistance by forming a nitride layer that acts as a barrier against corrosive elements. Finally, surface treatment enhances the fatigue strength of the mould steel by introducing local compressive residual stresses and reducing the risk of surface cracks and fatigue failure.

Two surface induction hardening tests were carried out at 950°C and 1050°C on 75x75x20 mm samples with a heating time of 4 to 5 seconds at a frequency was 22 kHz. In Figure 7(a), a hardening above 600 HV10 (approx. 55 HRC) is measured at the surface of both samples. The hardening depth is greater for the sample at 1050°C than at 950°C, extending from 3 to 5 mm, but undesirable surface cracks were also noticed on the 1050°C test samples. Gas nitriding test was carried out on a 75x75x20 mm sample at 540°C for 24 hours. In Figure 7(b), a hardening up to 873 HV0.3 (approx. 66 HRC) is measured at the top surface. The hardening depth is about 0.2 mm, which is typical for this type of grade and conditions.

Figure 7: Hardness profiles obtained after (a) surface induction hardening at 950°C and 1050°C, and (b) gas nitriding at 540°C during 24 h.
CONCLUSIONS

Besides providing new insights on retained austenite decomposition and microstructural evolution during the tempering stage, this work evaluates the basic mechanical properties and others application properties of a pre-hardened modified H11 grade with reduced contents of Cr, Mo and V designed for plastic moulding applications.

As the thickness increases, it is difficult to obtain a uniform cooling in the thickness direction, some amount of retained austenite is likely to form locally due to the slower quenching rates in the core of the plates. The sequence of retained austenite decomposition during tempering was clarified from in situ synchrotron X-ray diffraction: it remains stable during heating and holding at the tempering temperature, and during the final cooling, fresh and hard martensite is formed from retained austenite due to carbon depletion during the previous stages of the tempering process.

This study demonstrates the industrial feasibility of obtaining the desired properties up to a thickness of 130 mm, namely a uniform hardness between 430 and 460 HBW, high-tensile properties superior to 1400 MPa UTS and impact properties greater than 15 J in the transverse direction (ST) at 20°C. Plate thickness up to 250 mm will be tested in a second industrial campaign. Fine optimization of the alloying content and process parameters will also further improve the impact properties.

The good thermal conductivity, along with the compatibility of the new product with important workshop operations such as machining, polishing or surface treatment create a new prospective to consider this pre-hardened steel as a cost-effective alternative to H11 or H13 steels sometimes used for plastic moulding applications. In addition, the overall good balance between strength and toughness opens the door to the manufacture of many tools, dies, and machine parts used in mechanical engineering applications.

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REFERENCES


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