Original Article

Machining-induced grain refinement of AISI 4340 alloy steel under dry and cryogenic conditions

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\textbf{A R T I C L E  I N F O}

Article history:
Received 30 May 2019
Accepted 21 July 2019
Available online 7 August 2019

Keywords:
Cryogenic machining
AISI 4340 alloy steel
Globular particle

\textbf{A B S T R A C T}

Machining significantly influences surface integrity, metallurgical structure, grain size and thereby the mechanical, chemical and physical properties of the component. The presented investigation concentrates on grain refinement in turning of AISI 4340 alloy steel in dry and cryogenic turning. With cutting temperatures below 950 °C, the resulted surface layer consists of a microstructure with ultrafine white globular particles in all samples. A higher percentage of these particles was observed when using cryogenic flushing. It resulted in improved surface and subsurface properties in terms of ultrafine microstructure concentration, higher micro hardness, influence on the thickness and composition of the surface, and subsurface layers and thermal stability.

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1. Introduction

In recent years, researchers have shown great interest in investigating the effects of grain refinement achieved by using various techniques. The beneficial influence of grain refinement includes the increase of yield strength of the component [1], improving ductility of welded sections [2] and enhancing the strength and toughness of a material without changing its chemical composition [3]. Microstructural changes also occurred due to element diffusion as a result of the prolonged sintering time [4] and other processes such as thixo-welding of AISI D2 Tool Steel [5], semi solid joining of two parts of AISI D2 for microstructural evolution [6].

Grain refinement also is a result of severe plastic deformation (SPD) techniques. Estrin and Vinogradov [7] extensively reviewed the substantial amount of research reporting on refined grain structure by means of SPD. SPD has been defined as “any method of metal forming under an extensive hydrostatic pressure that may be used to impose a very high strain on a bulk solid without the introduction of any significant change in the overall dimensions of the sample and having the ability to produce exceptional grain refinement” [7]. The authors listed 20 basic and modern SPD techniques used in the manufacturing industry such as equal-channel angular pressing (ECAP), high-pressure torsion (HPT), accumulative roll bonding (ARB) and twist extrusion (TE). SPD methods are powerful techniques for producing refined grain structure.
whether at the bulk of the microstructure or at the surface of a component [7].

Machining-induced grain refinement of the surface and subsurface of a machined material can be considered as surface SPD [8] or transformational grain refinement (TGR) [1]. TGR was introduced by Hodgson et al. [9], who developed a thermomechanical process to produce ultrafine (1 μm) ferrite grains transformed from the austenite grain structure with promising enhanced properties. In machining, the thickness of the affected layer depends on numerous parameters such as the thermal and mechanical properties of the workpiece, the coolant used, cutting parameters, and the cutting tool material and geometry such as rake angle and nose radius [10]. The relevance of the related conditions is to use the advantage of the heat and strain generated during cutting, which will induce a severe plastic deformation at the surface and consequently alter the microstructure. However, the level of heat and strain needs to be controlled to achieve optimum results of the grain refinement so that no grain growth will occur due to high temperature. Hence, a cryogenic coolant can be used to control the temperature rise.

The application of cryogenics in machining for inducing grain refinement has been explored in recent years. It can be concluded that cryogenic cooling does contribute to the surface enhancement of a component, namely surface roughness, grain refinement, hardness, residual stress, fatigue life and phase transformation [11]. The cryogenic cooling process is also more effective as claimed by Musfirah et al. [12] than dry cutting for reducing tool wear, lowering the required cutting force, improving surface roughness, lessening de-formation of microstructure changes at the sub-surface level, and eliminating contamination of the machined part. Furthermore, the cryogenic will cause sudden cold of the chips to become hard and brittle, which enhanced the chip breakability during the machining process of AISI 4340 [13].

However, the study of machining-induced grain refinement at the machined surface of martensitic microstructures is not yet very advanced. Usually, supplied raw materials have been heat-treated to deliver the mechanical properties as required by the industry. Many alloy steels used in the automotive and machine tool industries comprise martensitic microstructures that have been quenched and tempered. The hardness of the as-quenched martensite is decreased by tempering to restore the ductility of the steel. Therefore, a separate surface hardening process is needed to produce the hard layer of a component to fulfil the industrial requirements.

In the present research, the main objective is to investigate how grain refinement could be obtained by machining process in cryogenic environment of AISI 4340 alloy steel. In this study, the microstructures at the machined surface and subsurface of martensitic alloy steel are analyzed by microstructural observation to clarify the mechanism of grain refinement through machining under dry and cryogenic conditions. The cutting temperature generated during the machining operation was recorded to examine its relationship with the resulted microstructure.

### 2. Experimental work

The tested material was AISI 4340 alloy steel in a quenched and tempered state, with a 100 mm diameter and 317 HB hardness. The chemical composition is given in Table 1. AISI 4340 is a high-strength alloy steel widely used in the automotive and machine tool industries.

The microstructure of the as-received raw material revealed that it is a typical lath-martensitic structure as shown in Fig. 1. The bar was turned on a CNC lathe machine with an ISO P CVD coated (TiCN and Al₂O₃) carbide insert grade. Cutting parameters are listed in Table 2. For cryogenic flushing, a flexible hose was connected to the LN tank and a copper pipe was used as a nozzle pointed to the clearance face of the insert. The distance between the nozzle edge and the cutting point was fixed at 2 cm. During the experiments, the cutting temperature was recorded using a NEC Avio thermal infrared camera, model InRec Thermo Gear, which had been positioned at a 1 m distance from the measuring point. The recorded measurements were analyzed with InRec Analyser NS9500 software to calculate the maximum temperature generated during cutting.

The microstructure of the work material, before and after machining, was observed using a BX51M Olympus optical microscope and Merlin Compact Zeiss field emission scanning electron microscope. The specimens were mounted in a Bake-lite powder, grounded and polished to a mirror surface before being etched in 2% Nital solution for 5–6 s. Nano-hardness values after machining are the averages of three readings at different positions of the specimens measured with a Shimadzu Dynamic DUH-211S ultra-microhardness tester using a 19.61 mN load and a 10 s dwell time.

### 3. Results and discussion

#### 3.1. The cutting temperature during machining

The sources for the generated heat during machining are the primary deformation zone at the shear plane, the secondary deformation zone at the chip-tool interface, and the tertiary deformation zone at the tool-workpiece interface. It is expected that the machined surface and subsurface microstructures and the mechanical properties will be affected by the produced heat, therefore, the measurement of cutting temperature is important. Fig. 2 shows the measured cutting temperatures during turning in dry and cryogenic flushing. The application of cryogenic LN during turning has effectively reduced the cutting temperature by a range of 35–55% compared to dry turning, especially at higher cutting speeds. The lower temperatures in cryogenic machining
Table 2 – Cutting conditions of the experimental test.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cutting inserts</td>
<td>CNMA 120404</td>
</tr>
<tr>
<td>Tool holder</td>
<td>DCLNR 2020K12</td>
</tr>
<tr>
<td>Rake angle (°)</td>
<td>−6</td>
</tr>
<tr>
<td>Lead angle (°)</td>
<td>−5</td>
</tr>
<tr>
<td>Cutting speed (m/min)</td>
<td>160, 200, 240</td>
</tr>
<tr>
<td>Feed rate (mm/rev)</td>
<td>0.3</td>
</tr>
<tr>
<td>Depth of cut (mm)</td>
<td>1.0</td>
</tr>
<tr>
<td>Coolant</td>
<td>Dry and cryogenic (LN)</td>
</tr>
</tbody>
</table>

are advantageous to reduce the machined surface alteration caused by thermal activity [10]. Literature suggest that excessive heat during machining will result in thermal damage at the machined surface and subsurface influencing of the surface quality, increase of the tensile residual stresses, besides reducing the dimensional accuracy of the workpiece [11].

In addition, using LN as coolant can control the heat generated due to the increase of cutting speed. Fig. 2 proved the ability of LN to control the amount of heat generated at the critical cutting zone and to decrease the cutting temperature even with the increase in cutting speed. In dry cutting the expected increase of cutting temperature with higher cutting speed can be observed influencing component quality due to thermal distortion.

The cryogenic cooling effect was found to be more effective at higher speeds. This can be associated with the chip morphology formed during the cutting process. At higher speeds, the chip becomes thinner and curlier. In addition, the chips become harder with improved breakability due to sudden cooling by the cryogenic [14]. This makes the penetration of LN into the chip-tool interface easier, hence reducing the temperature.

4. Microstructure and properties

4.1. Microstructure at machined surface and subsurface

The machining-induced surface layer consists of two main layers as mentioned by Kaynak et al. [10]: the refined grain layer (RL) and transition layer (TL) as shown in Fig. 3. These two layers have different characteristics in terms of mechanical properties and chemical composition compared to the bulk material. The properties and the thickness of the layers depend on many factors such as the temperature generated and the heating-cooling rate during machining, original grain size, and the original mechanical properties of the bulk material.

Fig. 4 compares the thickness of refined grain layer (RL) and transition layer (TL) produced after machining in dry and cryogenic environment. At a cutting speed of 160 m/min, the difference of RL and TL thickness between dry and cryogenic turning is comparatively low. However, significant
differences have been observed in the thickness when cutting at 200 m/min and 240 m/min, where cryogenic turning induced thicker refined layers than dry turning, but on the other hand, dry turning has a larger transition layer compared to cryogenic turning. During machining, a significant portion of the heat will be transferred into the machined surface. In this research, the LN supply has been injected to the clearance face, allowing enough heat to be generated and transferred to the machined surface before the surface is being rapidly cooled by the LN. The rapid heating-cooling cycle with cryogenic increases the size of the refined grain layer beneath the surface. In dry turning, the cooling rate is lower. Therefore, the refined or recrystallized grains under the surface experience grain growth due to the thermal effect of higher temperatures. Only the grains near the surface will experience a fast cooling rate by means of air convection, maintaining small and refined microstructure. The transition layer occurs when the heat transferred to the subsurface results in localized thermal softening, hence easing the process of plastic deformation [11]. The slower cooling rate in dry turning allows the heat to be absorbed deeper in the subsurface, whereas much faster cooling rates and higher heat transfer coefficients in cryogenic turning cause lower temperatures at the surface as well as beneath the surface, thus decreasing the transition layer achieved in cryogenic turning. A similar investigation also claimed that cryogenic cutting resulted in a smaller affected layer beneath the surface [11].

In cryogenic turning there is no significant variation in the depth of each layer when machining at different cutting speeds. In dry turning there are noticeable pattern in the depth of RL which is decreasing with increasing cutting speed, while in contrast the TL thickness become larger with increasing speed. It is concluded that cryogenic turning produced a consistent depth of induced surface layer even when machining at different cutting speeds. This is explained by the proper control of cutting temperature in cryogenic turning (Fig. 2). When machining under dry conditions, the natural air convection is used to bring the temperature down. At slower cutting speeds, a larger portion of generated heat has been transferred to the machined surface compared to machining at higher cutting speeds [15]. Since the temperature recorded when dry turning at 160 m/min was not too high, this reduced the effect of grain coarsening due to the thermal effect and allowed a deeper refined grain layer to be developed. At higher cutting speeds, especially at 240 m/min, the higher temperature causes grain growth to occur at a smaller depth where the cooling rate is not fast enough. This leads to smaller RL and larger TL.

Fig. 5 shows a FeSEM microstructure of machined cross-section at 50k× magnification. Ultrafine white globular particles with a diameter smaller than 200 nm could be
observed in dry and cryogenic machining. These particles, observed in dry turning, can be associated with M/A island particles, when heat builds up, and austenite is formed at the machined surface. During cooling, phase transformation occurs and the austenite is transformed into ferrite, martensite and martensite/austenite (M/A) structures as shown in Fig. 5(a). The M/A island usually exhibits a high hardness characteristic [16], but is also claimed to be a factor in the initiation of cracks and brittle fractures [17].

During cryogenic turning, the temperature generated never exceeded the austenitizing temperature (Fig. 2). However, the produced heat was high enough to cause recrystallization at the machined surface. At the very high cooling rate, a tempered/recrystallized martensitic structure was observed at the very edge of the machined surface, with homogeneously distributed ultrafine white globular carbide particles within the martensite matrix as shown in Fig. 5(b). This is supported by the theory suggested by Shokrani et al. [18] that using cryogenics enhances the precipitation of carbide particles into the martensite matrix, promotes the refinement of the carbide particles and produces a more homogeneous ultrafine carbide distribution and increases hardness [19].

4.2. XRD spectra of machined surfaces

The XRD measurements were conducted to verify whether the retained austenite phase in the as-received material microstructure was transformed during machining. The XRD peaks of all machined surfaces shown in Fig. 6 provide the evidence of phase transformation. The (200) austenite peak and (200) ferrite/martensite peak at approximately 10° and 65° respectively, do not exist on all XRD graphs of the machined surfaces. The (110) ferrite/martensite peak at 44° is the only peak left on all the machined surfaces. This indicates that the machined surfaces have gone through a phase transformation to fully martensite with only one crystallographic orientation. The continuous-cooling transformation (CCT) phase diagram of AISI 4340, as generated by IMATPRO 4.0, confirmed that if the machined surface has been cooled at a considerably fast rate, the cooling curve will pass in front of the ferrite and bainite noses, hence resulting in the formation of only a martensite phase. The alloying elements in AISI 4340 cause a shift of the isothermal (TTT) and CCT diagrams, reducing the martensite start (Mₜ) and martensite finish (Mₐ) time, therefore increasing the hardenability of the steel [20]. Besides the elimination of retained austenite peak, all machined surfaces also showed the broadening of the (110) martensite peak width and the peak shifting to a lower angle compared to the as-received material (Fig. 7). The broadening of (110) the martensite peak width at approximately 45° in machined surfaces is associated with the finer average crystal sizes and the high strains due to strain localization that occurred during the machining [21]. This is parallel with the grain refinement observed at all machined surfaces as shown in Fig. 5.
4.3. Hardness of the machined surface and subsurface

The effect of grain refinement is usually associated to hardness, as stated in Jawahir et al. [19]. According to Kaynak et al. [10], increased hardness at the surface and subsurface was contributed by several reasons, such as reduction of thermal softening effect, strain hardening, twinning deformation and grain refinement. Fig. 8 shows the hardness profile of the machined surface and subsurface in dry and cryogenic environments at three different cutting speeds. It is expected that the machined surface is harder compared to the bulk and the hardness is lower with increasing profile depth [22].

Cryogenically machined surfaces show a superior value of hardness compared to that of dry machined surfaces in all speed variations. This might be due to the reduction of the thermal softening effect and more severe strain hardening or work hardening at low temperatures [23], which resulted in higher density of refined carbide particles in cryogenically machined surfaces. During or after plastic the deformation process, if the temperature rises up to the restoration point of the material, the effect of work hardening will be weakened. Hence, by applying LN during the machining process, the temperature rise can be prevented and the positive effect of work hardening on the machined surface can be maintained.

5. Conclusion

The microstructure of the machined surfaces of the AISI 4340 martensitic alloy steel generated under different turning conditions were investigated. It can be concluded that cryogenic turning provides the advantage of controlling the temperature rise during a machining process; hence, the quality of the machining-induced surface layer was preserved even
in turning at different cutting speeds. Ultrafine white globular particles were formed and improved the strength and hardness of the machined surface. Higher density and more homogeneous ultrafine white globular particles, which are associated with the carbide precipitates, were observed in all cryogenically machined surfaces, which contributed to the higher hardness compared to those machined under dry conditions.

**Conflicts of interest**

The authors declare no conflicts of interest.

**Acknowledgements**

This project is supported by Government of Malaysia and Universiti Kebangsaan Malaysia under DIP-2018-026 and GUP-2017-048 Grants.

**References**


