Machining Heat Induced Phase Transformation on the Surface Hardening Layer of High Strength Ferrous-Based Biomedical Stainless Steel

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The present study investigates the drilling characteristics of a high-strength Fe–8Al–30Mn–1C–1Si–3Cr (mass%) biomedical alloy. After machining, a surface-hardening layer with a Vickers hardness number (H_V) equal to 600 was observed. In addition, a $\gamma \rightarrow (\gamma + \kappa)$ phase transition was observed in the matrix and at the stress-induced twin boundaries of the surface-hardening layer. κ -phase carbides ((Fe,Mn)₃AlC_x) having an L'_{12} structure with the lattice parameter $a = 0.375$ nm were precipitated. Furthermore, the heat transfer coefficient of the present alloy was 0.083 cal/(cm² s °C), which was lower than that of AISI 304 stainless steel, which was 0.098 cal/(cm² s °C). The instantaneous cutting temperature of the present alloy was approximately 650°C during the machining process. It is believed that the formation of κ -phase carbides not only decreases the machinability of the present alloy, but also reduces the life of the cutting tool. These features could be useful in further understanding the relationship between the machinability and the microstructure of Fe–Al–Mn–C-based alloys, and thus provide information that would be allow these alloys to be used in biomedical and industrial applications. [doi:10.2320/matertrans.M2012107]

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1. Introduction

Austenitic Fe–Al–Mn–C alloys generally have optimal chemical compositions in the range Fe–(4.9–11.0)Al–(23.7–35)Mn–(0.5–1.5)C (mass%) and are formed by conventional casting with or without subsequent plastic deformation.^{1–5)} Al is added to promote resistance to corrosion and oxidation by the formation of a continuous protective layer of Al₂O₃ on the surface of the alloys.^{2,6,7)} Mn is an austenite (γ phase) structure stabilizer,⁸⁾ and carbon is an important strengthener. Using between 0.5–1.5 carbon (mass%) could lead to the coherent precipitation of fine κ -phase carbides ((Fe,Mn)₃AlC_x) with an L'_{12} structure within the γ matrix during the aging process. These precipitates have a strengthening effect.^{9,10)} Thus, they have been developed as a promising alternative to certain conventional biomedical Fe–Cr–Ni-based stainless steels, as they replace Cr and Ni with lighter and cheaper elements such as Al and Mn without sacrificing desirable mechanical and biological properties.

Moreover, high-temperature corrosion and oxidation resistance as well as strength can be enhanced with metallic elements such as Si, Cu, Ti, Mo, Co and Cr.^{3–5,11–14)} Of these, Si and Cr have been found to be the most effective in improving resistance to high-temperature corrosion and oxidation and environmental embrittlement.^{12,15)} As mentioned above, Fe–Al–Mn–C–X (X = Si, Cu, Ti, Mo, Co or Cr) alloys have numerous attractive properties such as low density, high strength, superior ductility, good casting ability and good atmospheric corrosion resistance.^{12,16,17)} Hence, Fe–Al–Mn–C–X alloys are most suitable for the automotive, shipbuilding, and golf industries. Moreover, Chen *et al.*¹⁸⁾ have shown that a nanostructured oxide layer was formed

on the Fe–Al–Mn alloy surface after electrical discharge machining; this layer could increase alloy biocompatibility. Thus, Fe–Al–Mn and Fe–Al–Mn–C–X alloys can also be developed as potential biomedical alloys.

Nevertheless, in the case of industrial and biomedical applications, the machinability of materials is significantly associated with the manufacturing process. Chao *et al.*¹⁹⁾ have reported that in the case of Fe–(4–12)Al–30Mn–1C (mass%) alloys, produced after the tuning of different parameters, the surface roughness was in the range of 0.8–2.7 μ m. The feed rate has a significant effect on the surface roughness of these alloys. However, there is a lack of information about the drilling characteristics of Fe–Al–Mn–C–X alloys. Therefore, the purpose of the present study was to investigate the drilling characteristics of an Fe–8Al–30Mn–1C–1Si–3Cr (mass%) alloy with a pure γ phase structure, and to elucidate and discuss the mechanism responsible for the difficulty encountered in machining the alloy, while providing information that could help in the use of such alloys in industrial and biomedical applications.

2. Experimental Procedures

An Fe–8Al–30Mn–1C–1Si–3Cr (mass%) alloy sample was prepared in a vacuum induction furnace with a nitrogen atmosphere using AISI 1008 low carbon steel, 99.7% pure electrolytic Al, 99.9% pure electrolytic Mn, pure carbon powder, pure silicon and pure chromium. After melting and stirring, the alloy was poured into investment casting molds (with a diameter of 80 mm and length of 300 mm) preheated to 1150°C and its chemical composition, determined by inductively coupled plasma-atomic emission spectrometry, was found to be Fe–7.86Al–29.75Mn–0.95C–0.94Si–2.88Cr (mass%). The obtained ingots were then homogenized at

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Table 1 The drilling parameters and AMN of holes that could be drilled in the alloys with various drill diameters.

Drills	Diameter (mm)	Drilling parameters			Trial materials		
		Cutting velocity (m/min)	Feed rate (mm/rev)	Cutting depth (mm)	AISI 304 SS	S45C CS	The present alloy
					AMN of drilling holes		
HSS	$\phi 3$	3.46	0.185	15	38	48	30
	$\phi 6$				30	42	25
	$\phi 9$				22	34	16
	$\phi 12$				13	18	6
HSS-CN	$\phi 3$	3.46	0.185	15	48	48	37
	$\phi 6$				36	48	29
	$\phi 9$				27	40	20
	$\phi 12$				19	32	10

1200°C for 4 h in a protective argon atmosphere and hot-forged into bars with a diameter of 40 mm and length of 100 mm. Subsequently, the oxidation layers covering the bars were machined away. Before the tests, the bars were cut into smaller pieces that had a diameter of 40 mm and length of 25 mm. They were then solution heat-treated at 1050°C for 40 min (quenched rapidly in water). Therefore, the specimens had a pure γ phase structure at room temperature.

The drilling test was carried out on an upright drilling machine (MFG type, Tay Shing) using high-speed tool steel (HSS) and HSS-coated carbon nitride (HSS-CN) drills. The helix angle and lip angle of the drills were 32 and 118°, respectively. The drilling parameters are presented in Table 1. The average maximum number (AMN) of holes that could be created with 3-, 6-, 9- and 12-mm-diameter drills under suitable drilling conditions were determined in order to evaluate the drillability. Drilling tests were also performed on AISI 304 stainless steel (SS) and S45C carbon steel (CS) blocks with dimensions of 20 × 20 × 20 mm for comparison. The average of the values for five tests was used.

The microhardness measurements were made with a standardized Vickers microhardness tester (MVK-100, Akashi) using 300 g load and a dwell time of 10 s. The average of the values obtained for ten tests was used. Moreover, the microstructural characteristics of the cut surface of each specimen were identified using a JEOL-200CX transmission electron microscope (TEM) operated at 200 kV. Thin foils were polished to a thickness of around 40 μm , and subsequent electropolishing was performed using an electrolyte containing 60% ethanol, 30% nitric acid and 10% acetic acid at a current density of $(1.5\text{--}2.5) \times 10^4 \text{ A/m}^2$ at temperatures less than -10°C . A thermal conductivity analysis was conducted using an infrared thermography device (TVS-100 series, Handy Thermo).

3. Results and Discussion

Figure 1 shows a bright-field (BF) electron micrograph of the alloy that had been solution-treated at 1050°C for 40 min. The micrograph was recorded from the matrix in the [001] zone, indicating that some dislocations were formed in the matrix. A selected area electron diffraction pattern (SAEDP)

Fig. 1 BF electron micrograph of the alloy that had been solution-treated at 1050°C for 40 min, which was taken from the matrix in the [001] zone.

Fig. 2 The values of the average surface hardness near the holes in the investigated specimens drilled by the 9 mm HSS drill.

also revealed that the matrix has a single γ phase and had a face-center-cubic structure with the lattice parameter $a = 0.375 \text{ nm}$. No other precipitates were found within the γ matrix of the as-quenched alloy. This microstructure is the same as that reported for an Fe-8.7Al-28.3Mn-1C-5.5Cr alloy by Liu *et al.*¹⁾

In the drilling tests, the AMN of holes that could be drilled in the alloys with various drill diameters is given in Table 1. The AMN of holes drilled in the S45C CS, AISI 304 SS and present alloy using the 3 mm HSS drill were 48, 38 and 30, respectively. The AMN decreased with an increase in the drill diameter. Only six holes could be drilled in the present alloy with a 12 mm HSS drill. Obviously, the drillability of the alloys when the HSS-CN drills were used was similar to that observed for the HSS drills. The AMN could be increased by around 4–10 holes by using the HSS-CN drills. The results significantly demonstrate that the Fe-8Al-30Mn-1C-1Si-3Cr alloy is more difficult to drill than AISI 304 SS and S45C CS. Furthermore, it was found that the drill life when the drills were used on the present alloy was approximately 78.9 and 62.5% of that for AISI 304 SS and S45C CS, respectively.

Figure 2 displays the values of the average surface hardness near the holes in the investigated specimens drilled by the 9 mm HSS drill. It was found that the present alloy had

Fig. 3 TEM images taken from the hardened surface layer of the present alloy: (a) BF image showing a high density of dislocations; (b) BF image showing stress-induced twins (foil normal is $[011]$; $hkl = \gamma$ matrix); (c) higher magnification BF image taken from the stress-induced-twin boundary in the $[111]$ zone ($hkl = \kappa$ phase carbide, $hkl = \gamma$ matrix); and (d) $\vec{g} = 01\bar{1}$ κ phase DF image.

a 0.2 mm thick hardened surface layer, whereas S45C CS and AISI 304 SS each had a layer that was 0.3 mm thick. Nevertheless, the Vickers hardness number (H_V) of the layer on the present alloy, which was approximately 600, was higher than those of the layers on S45C CS or AISI 304 SS. In addition, we also found that in the present alloy, the drilled holes expanded by 0.02–0.05 mm in diameter. These features, namely, the hardened surface layer and the expansion of the holes were observed only in the alloy specimens drilled by 9 and 12 mm HSS-CN drills. As for the alloy specimens drilled using a drill with a smaller diameter (<6 mm), both the hardened surface layer and the increase in the hole sizes were not observed.

In order to elucidate the mechanism responsible for the difficulty encountered in machining the present alloy, the specimens with hardened surface layer were analyzed using TEM. Figure 3(a) is a BF electron micrograph of the hardened surface layer of the present alloy. It can be clearly seen that a large number of dislocations were formed in the layer. Moreover, Fig. 3(b), which is a foil normal $[011]$ zone axis BF electron micrograph of the hardened surface layer of the present alloy, reveals that stress-induced-twins were also formed in the layer.²⁰⁾ Furthermore, some small black particles (as indicated by the arrows) were found to have precipitated within the γ matrix and stress-induced twin

boundaries. Figure 3(c) is a magnified BF image of the stress-induced twin boundary along the $[111]$ zone axis. It reveals that in addition to the reflection spots of the γ phase, the SAEDP also comprises small superlattice spots. On the basis of the camera length and the d -spacings of the spots, we identified these small black particles as κ -phase carbides ($(\text{Fe,Mn})_3\text{AlC}_x$) having an $L'1_2$ structure with the lattice parameter $a = 0.375$ nm.^{9,10,21,22)} Figure 3(d) is a $\vec{g} = 01\bar{1}$ κ -phase dark field (DF) electron micrograph that shows clearly the presence of the κ -phase within the γ matrix and the stress-induced twin boundaries.

Some of the experimental results obtained are discussed in the following section. As mentioned above, the κ -phase carbides were noticed to have precipitated on the hardened surface layer of the present alloy. Many researchers have reported that the κ -phase carbides precipitate within the γ matrices of Fe–Al–Mn–C and Fe–Al–Mn–C–X alloys after aging at 450–750°C.^{3,4,23–25)} However, there have been no prior studies reporting κ -phase carbide precipitation by machining or any other material-working process. Thus, this implies that the heat produced during the cutting process (or “cutting heat”) plays an important role in the formation of the κ -phase. In order to elucidate the formation mechanism of the κ -phase, thermal conductivity analyses of the present alloy and AISI 304 SS were performed. Figure 4 shows the

Fig. 4 Thermal distribution images of (a) AISI 304 SS and (b) the present alloy heated at 120°C for 5 min.

thermal distribution images of AISI 304 SS and the present alloy; samples of both materials were heated at 120°C for 5 min. It is clear that the surface temperature of the present alloy was lower than that of AISI 304 SS. The heat transfer coefficients of the present alloy and AISI 304 SS obtained from the infrared thermography device were 0.083 cal/(cm²s °C) and 0.098 cal/(cm²s °C), respectively. These results reveal that the present alloy has slightly lower thermal conductivity. Therefore, the cutting heat produced between the nose of the tool and the cutting surface could easily have built up to a large value during the machining process. Moreover, we found that the instantaneous cutting temperature of the present alloy was approximately 650°C, which is equivalent to the aging temperature. Hence, it can be reasonably deduced that the κ -phase carbides were formed within the surface hardening layer of the present alloy. Accordingly, a $\gamma \rightarrow (\gamma + \kappa)$ phase transition was observed in the matrix and at the stress-induced twin boundaries after the machining process. It is believed that the formation of κ -phase carbides not only decreases the drillability of the present alloy but also reduces the life of the cutting tool.

4. Conclusions

- (1) The drill life with respect to the present alloy was approximately 78.9 and 62.5% of that for AISI 304 SS and S45C CS, respectively. In addition, the present alloy had a 0.2 mm hardened surface layer and a Vickers hardness number (H_V) approximately equal to 600.

- (2) A large number of dislocations, stress-induced twins and κ -phase carbides ((Fe,Mn)₃AlC_x) having an L'1₂ structure with the lattice parameter $a = 0.375$ nm were formed within the surface hardening layer of the present alloy after the machining process.
- (3) The heat transfer coefficients of the present alloy and AISI 304 SS were found to be 0.083 cal/(cm²s °C) and 0.098 cal/(cm²s °C), respectively. The instantaneous cutting temperature was approximately 650°C for the present alloy.

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