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Effect of Forging Temperature on Biodegradable Mg-0.7%Ca Alloy Properties for Implant Application

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Abstract. The potential of binary Mg-Ca alloy as biodegradable material is considerable interest in implant application among researchers. This research was conducted to investigate the effect of different forging temperature and forging speed on the hardness, microstructure and corrosion rate of Mg-0.7%Ca. The experiment was established by preparing the alloy sample with 0.7%wt calcium content. The forging process was carried out under four different temperature variations of 140°C, 180°C, 220°C, and 260°C (±10°C) with two different speed;25 and 45 strokes per minute (spm). The samples microstructure was examined by optical microscope and scanning electron microscope (SEM) equipped with energy dispersive X-ray (EDX). The mechanical properties of the forged samples were measured in its hardness and plastic deformation ability along with samples cold-working percentage. The corrosion rate was determined by performing the electrochemical test in simulated body fluid. This research found that increases of forging temperature and forging speed provide a higher rate of recrystallization and Mg2Ca compound precipitation results in greater hardness, increase deformation and reduce the cold-working percentage. However, the investigated factors still led to a high corrosion rate compared to a previous study and consequently, reduce the feasibility of the alloy in implant application for biodegradable material.

Keywords: forging, biodegradable, implant, calcium, temperature, microstructure

1. Introduction

The research involves Mg-Ca binary alloys as biodegradable materials have attracted various investigation due to their biocompatibility feature and mechanical properties that exhibit similarities to natural bone [1]. As binary Mg-Al and Mg-RE (rare earth element) alloys are substantially harmful that could cause nerve toxicity due to Al, and hepatotoxicity for the latter [2][3], Mg-Ca alloy is favourable since it has a similar density to the bone and shown to be degraded within the bone and biocompatible in vivo and vitro [4][5][6]. Exclusive of the stated qualities, Mg-Ca binary alloy having less than 3% of calcium content is fairly an adequate threshold for implant application [4,3]. The forging process has been proposed with increasing the forging temperature to reduce the critical shear stress stored during cold-working [7]. Previous researchers found that increasing forging temperature on Mg-1%Ca alloy result in higher corrosion rate due to high Mg_2Ca precipitation thus lead to faster degradation of the implant before the bone healing process can be achieved [7]. In order to decrease the corrosion rate of Mg-Ca alloy, an investigation is conducted by reducing the Ca addition at 0.7%, decreasing forging temperature and speed than previous study [7] to improve the corrosion quality along with the mechanical properties of Mg-Ca alloy and thus, increase the feasibility of the alloy in implant application by forging process technique.

2. Experimental Procedure

2.1. Material Fabrication

The process on preparing the Mg-0.7%Ca alloy involves gravity casting whereby commercial purity magnesium ingot (99.9%) and Mg-30wt%Ca master alloy was melted in a mild steel crucible under the temperature of 670°C in an induction furnace protected with a continuous flow of argon gas to avoid oxidation. The molten alloy was then poured into a 300°C preheated simple steel mould. The samples were obtained from the middle of the cast product by the mechanical cutting process.

2.2. Forging Process

The forging process was performed using AIDA NC200 hot forged stamping machine (200 tonnes) whereas the speed was regulated at 25 and 45 strokes per minute (spm) under 1962kN constant force. The alloy was forged at the temperature of 140 °C, 180°C, 220°C and 260 °C (+10 °C). Forged samples were 61 x 22 x 26.5 mm.

2.3. Microstructure Evaluation

The microstructure of the alloys was characterise using X-ray diffractometer (CuKα radiation) for identification of the crystal structure of the phases. Optical microscope was used to characterise the surface morphology while the chemical composition was analysed energy diffractometry spectrum (EDS).

2.4. Hardness Measurement

The hardness of the samples was tested as-cast and as-forge using Matsuzawa DVK-2 material testing machine, at nine different locations of the samples according to ASTM-E98-82 (Standard test method for Vickers hardness of metallic materials 2003). The samples were cut in 10 x 15 x 15 mm dimension.

2.5. Corrosion Test

The corrosion of the forged samples was evaluated using a potentiostat/ galvanostat corrosion test system (Parstat-2263) according to ASTM G102-89. A saturated calomel electrode (SCE) and graphite were used as the reference and counter electrodes, respectively. 500 ml SBF was used as the test solution and the temperature of the test was maintained as 37˚C during the experiment.

3. Results and Discussion

3.1. Microstructure Characterisation

The production of Mg-0.7%Ca was successfully prepared as described in its chemical composition in Table 1 with EDX analysis in Figure 1 whereas 0.7% of Ca is presented in the binary alloy composition. As shown in Figure 2 and 3, the grain size of the as-cast Mg-0.7%Ca is smaller compared to 99.9%Mg. The microstructure of the forged samples under different temperatures and speed are displayed in Figure 4 and 5. Both figures shows that better grain size reduction is observed at increasing forging temperature except at 220°C and 260°C whereby the grain starts to grow due to the forging temperatures are in the range of recrystallization or warm-working condition and hence, a low strain-hardening was subjected to the alloy creates lesser dislocation for blocking the growth of grains. Low forging speed, on the other hand, is also a factor to be considered whereas the grain, in which already had lesser dislocation, have more time and fewer blockage for the grain to grow along the surface. The microstructure also described twinning planes distribution in the grains. This indicates that twinning system is the dominant deformation factor instead of the slip system. Increasing in forging temperatures provide more driving force of recrystallization but, without sufficient forged speed, the grain size will grow larger due to low strain energy and deformation. Apart from that, precipitation of Mg_2Ca phase in α(Mg)primary grain and grain boundaries can be observed in both Figure 4 and 5 with most on higher forging speed. The SEM images in Figure 6 indicates that the precipitation increase with increasing on forging temperature and the speed as verified in Figure 7 on the identification of Mg_2Ca precipitation by EDS analysis. The precipitation was detected at 2Θ = 33.797° corresponding to (1 1 2) crystallographic planes in both as-cast and forged samples, as shown in the XRD pattern in Figure 8. The results show that the presence and formation of α(Mg) matrix and Mg_2Ca phases are in all samples, which mean that Ca is found in the form of Mg_2Ca compound. in the amount of Mg_2Ca phase in the alloy

Table 1. Chemical composition of Mg-0.7%Ca.

Element	Wt (%)	At (%)
MgK	99.29	99.61
CaK	0.71	0.39

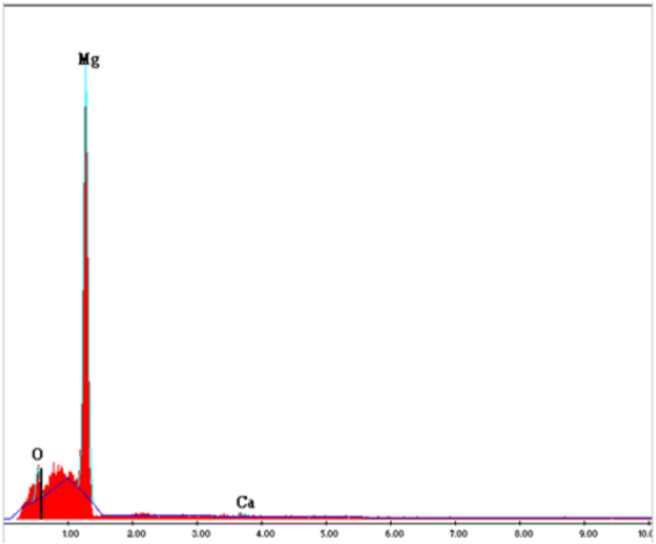


Figure 1. EDX analysis of Mg-0.7%Ca.

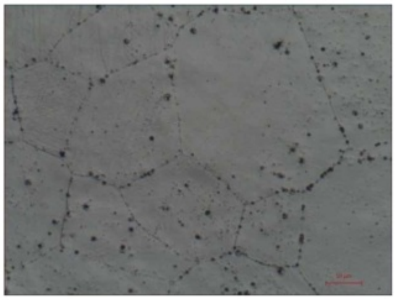


Figure 2. Microstructure of pure 99.9%Mg (50x magnification).

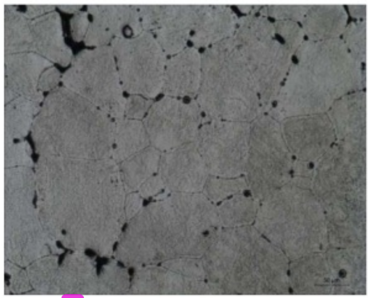


Figure 3. Microstructure of Mg-0.7%Ca (50x magnification).

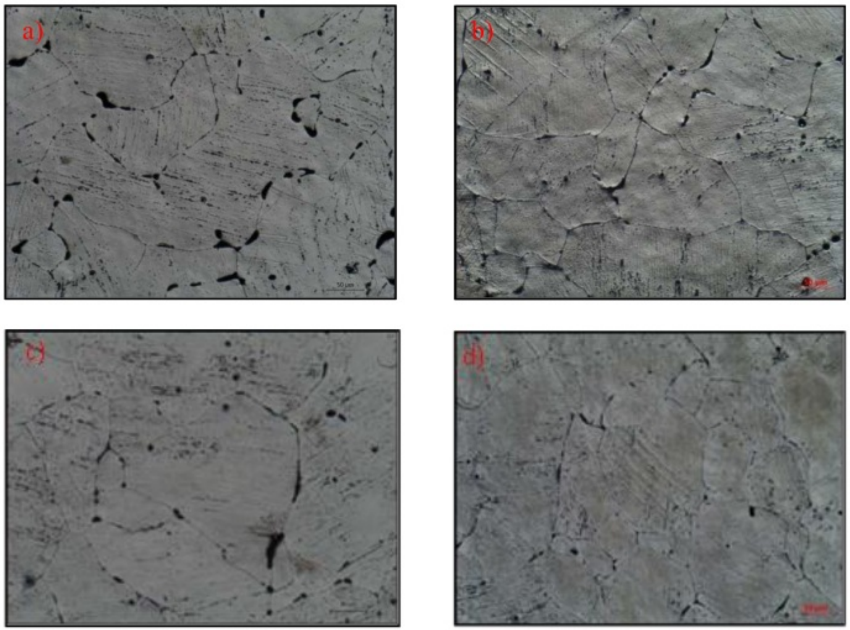
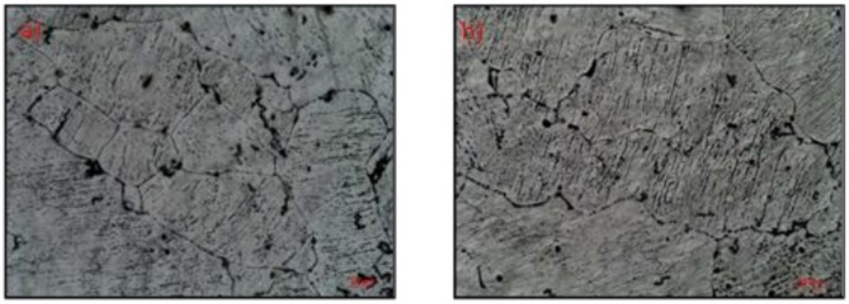


Figure 4. The microstructure of Mg-0.7%Ca alloy forged at (a) 140ºC, (b) 180ºC, (c) 220ºC, and (d) 260ºC at the speed of 25spm.



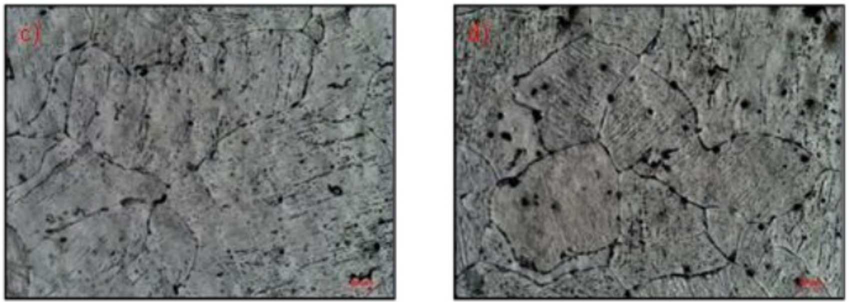


Figure 5. The microstructure of Mg-0.7%Ca alloy forged at (a) 140ºC, (b) 180ºC, (c) 220ºC, and (d) 260ºC at the speed of 45spm.

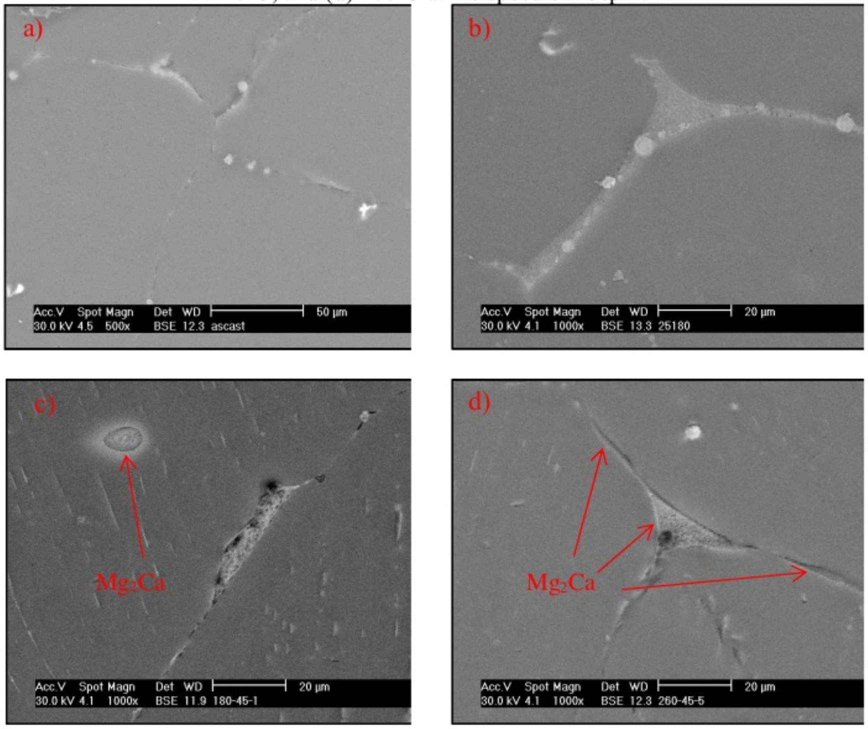


Figure 6. SEM images (a) as-cast, and forged samples at (b) 180ºC (25spm), (c) 180ºC (45spm) and (d) 260ºC (45spm).

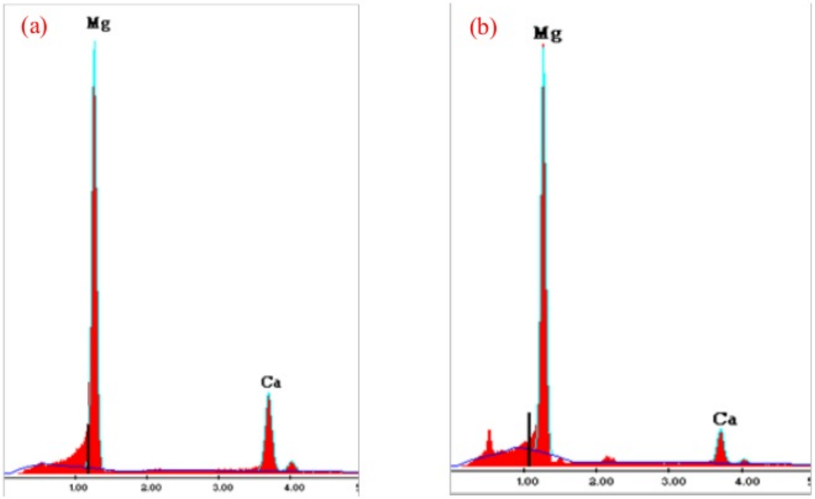


Figure 7. EDX analysis of Mg_2Ca at sample (a) 180°C at 45spm and (b) 260°C at 45spm.

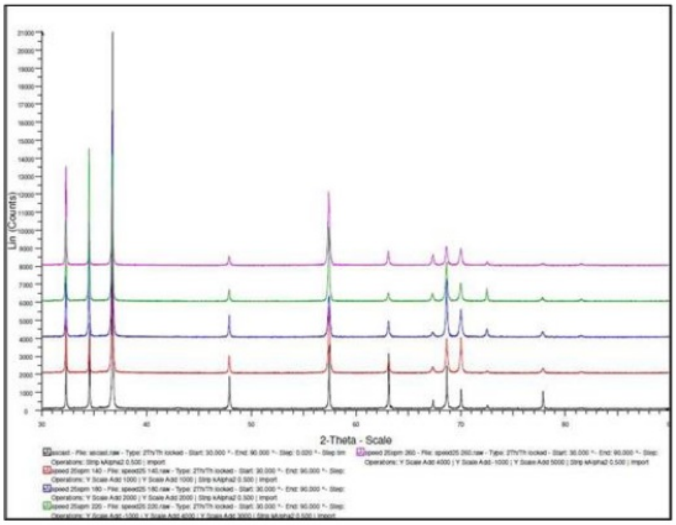


Figure 8. The XRD patterns of the as-cast and as-forged samples at different temperature for speed 25spm.

3.2. Mechanical properties

The hardness of 99.9%Mg sample was measured as 28.9HV while as-cast Mg-0.7%Ca alloy was at 36.2HV. Ca improves the mechanical property of Mg. Table 2 and Figure 9 show the Vickers hardness of forged samples under 25spm and 45spm, respectively for four types of temperatures. The hardness increasing when forged temperature and forged speed increase due to grain refining under recrystallization condition. However, there was a sudden trend drop in value, starting at 220°C until 260°C for both forging speeds. Higher forging temperature in which already in the recrystallization temperature range, decrease the hardness of samples as a result of Mg_2Ca migration into grain boundaries and dissolution in α(Mg) matrix. Therefore, grain size starts to grow faster after recrystallization phase. High forging speed and temperature affect the increase in the deformation of the binary alloy, as shown in Table 3 and graphically visualised in Figure 10. The pronounce of forging speed effects is clear since high force is necessary in cold-working in order to drive the dislocation movement. Cold-working percentage is decreased by the increasing of forging temperature and slower forging speed, as shown in Table 4 and Figure 11. The dislocation density in a metal increases with deformation, due to dislocation multiplication or the formation of new dislocations. As the dislocation density increases, the resistance to dislocation motion by other dislocations becomes more pronounced. Thus, the imposed stress necessary to deform a metal increases with increasing of cold-working. In the recrystallization phase, the mechanical properties increment during cold-working are restored results in weaker material but enhance ductility. Besides that, the rate of recrystallization increases when the cold-working percentage is higher.

Table 2. Vickers hardness of forged Mg-0.7%Ca.

Temperature (°C)	Hardness value (HV)	
	25spm	45spm
140	40.7	43.7
180	42.9	47.7
220	41.0	45.0
260	40.0	44.0

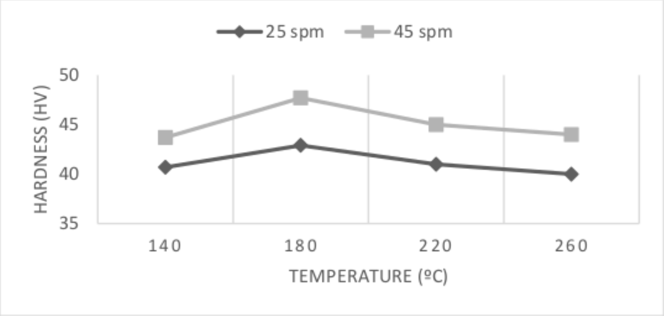


Figure 9. Graph of Vickers hardness of forged Mg-0.7%Ca versus temperature and forging speed.

Table 3. Deformation value of forged Mg-0.7%Ca.

Temperature (°C)	Deformation (mm)	
	25spm	45spm
140	23.30	23.79
180	23.50	24.00
220	23.80	24.30
260	24.20	24.60

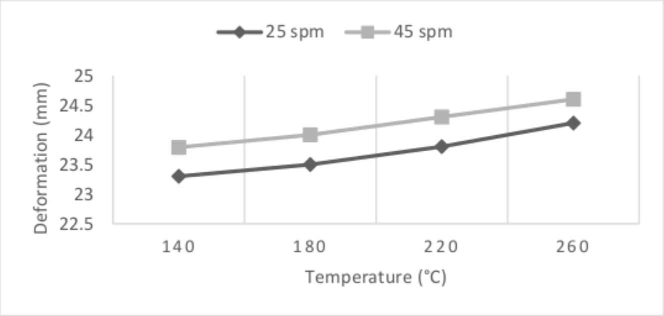


Figure 10. Graph of deformation of forged Mg-0.7%Ca versus temperature and forging speed.

Table 4. Cold-working percentage.

Temperature (°C)	Cold-working percentage (%)	
	25spm	45spm
140	2.80	1.25
180	2.25	0.79
220	1.20	0.81
260	0.50	0.41

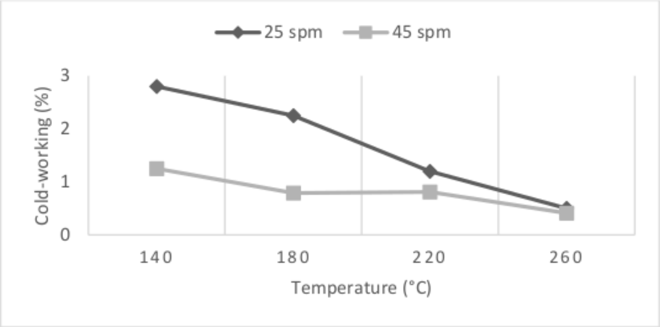


Figure 11. Graph of cold-working percentage versus temperature and forging speed.

3.3. Corrosion electrochemical testing

The potentio-dynamic polarisation curves of the as-cast Mg-0.7%Ca alloy and the forged samples under different temperatures for 25spm and 45spm of forging speed have been obtained and shown in Figure 12 for the latter. The derived current density (I_{corr}), corrosion potential (E_{corr}) and corrosion rate are given in Table 5 and graphically displayed in Figure 13. The corrosion rate is seen to be decreased when subjected to higher forging speed. The increasing in forging temperature increases the corrosion rate but sudden drop starting with 220°C for both forging speeds. However, at 260°C, corrosion rate spikes under 45spm forging speed meanwhile at 25spm, the trend still shows a drop. The precipitation of Mg_2Ca increase the corrosion rate [7] which is true for the samples measured at 180°C and 260°C, but both samples have different grain refinement whereby the samples forged at 180°C is harder due to smaller grain size whereas samples forged at 260°C experienced grain growth thus vulnerable for corrosion to take place.

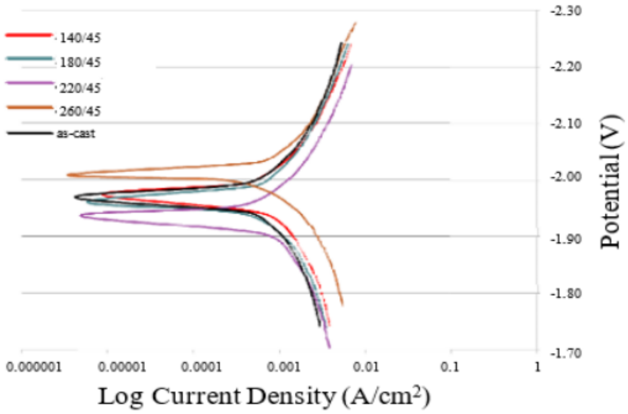


Figure 12. Tafel curves of as-cast and as-forged samples at speed of 45spm

Table 5. Chemical composition of Mg-0.7%Ca.

Sample	E_{corr} (mV)	I_{corr} (µA) x10^3	Corrosion rate (mmpy)
as-Cast	-1969.80	5.63	129
as-forged 140/25	-1978.27	6.53	149
as-forged 180/25	-2009.39	1.05	239
as-forged 220/25	-2010.31	7.70	176
as-forged 260/25	-1962.39	5.67	129
as-forged 140/45	-1974.05	5.27	120
as-forged 180/45	-1961.57	6.83	156
as-forged 220/45	-1937.60	5.92	135
as-forged 260/45	-2008.23	458.00	10500

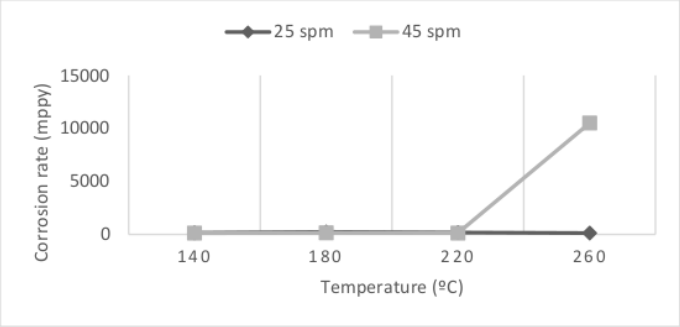


Figure 13. Graph of corrosion rate of forged Mg-0.7%Ca versus temperature and forging speed

4. Conclusion

The following conclusion can be drawn based on these research findings:

- Forging temperature increase the rate of recrystallization, which generates equiaxed and finer grains in Mg-0.7%Ca except when the temperature rose started from 220°C, grain size tends to grow and largely under low forging speed.
- High forging speed and temperature increase the precipitation of Mg_2Ca, which results in higher corrosion rate and deformation. However, the effect of both factors on hardness was different, whereby Mg-0.7%Ca forged at 180°C gives better hardness value than 260°C. This shows that forging speed and temperature are both significantly affect the alloy properties.
- Cold-working percentage is decreased when subjected to higher forging temperature and speed, thus improves material hardness.
- The corrosion rate value of Mg-0.7%Ca obtained in this research is not far from a previous study [7] on Mg-1%Ca although the forging temperature and speed used are lower. This shows that the forging process does not provide the intended corrosion quality for this alloy.

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