行政院國家科學委員會專題研究計畫 成果報告

含釩超輕鎂鋰合金之性質與表面處理研究
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計 畫 主 持 人 ： 吳泓瑜

計畫參與人員： 碩士班研究生-兼任助理人員：林家宇
碩士班研究生-兼任助理人員：陳宏偉
碩士班研究生-兼任助理人員：徐維謙
碩士班研究生-兼任助理人員：林峰正
碩士班研究生-兼任助理人員：鄒明達

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行政院國家科學委員會專題研究計畫成果報告

含鈧超輕鎂鋰合金之性質與表面處理研究

Study of the properties and surface treatment of ultra light Mg-Li Alloys containing Sc

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計劃主持人: 吳泓瑜　　中華大學機械與航太工程研究所教授

E-mail: ncuwu@chu.edu.tw

計畫參與人員：林家宇、陳宏偉、徐維謙、林峰正、鄒明達　中華大學機械與航太工程研究所研究生

中文摘要

本研究以含10wt%鋰的鎂鋰合金為基材，主要以添加Al、Zn及微量的Sc元素等來改善Mg-Li 合金之機械性質，並研究鈧對鎂鋰鋁鋅合金之影響。實驗設計包括時效硬化處理、微結構觀察、硬度測試等方法，並使用X光繞射分析儀進行不同處理過程微觀結構中相之鑑定，根據其結果加以分析；同時也以冷壓延探討Sc對鎂鋰合金加工硬化的影響。實驗結果顯示，含Sc鎂鋰合金僅具有自然時效之硬化效果，在高於室溫之時效處理中，硬度會快速的降低。冷作加工前，將擠製件進行退火熱處理，有助於提升加工硬化的效果。

關鍵詞：鎂鋰合金、X光繞射分析、時效硬化、加工硬化

Abstract

An Mg–Li based alloy containing Sc addition has been prepared by melting and solidification in a carbon steel crucible, and extruded at a billet preheating temperature of 200°C with an extrusion ratio of 28. Age heat treatments and thermomechanical processing were performed to investigate the effect of minor addition of Sc on the microstructures and mechanical properties. Hardness, optical microscopy, X-ray diffraction studies, and tensile tests were carried out to explore the variations in microstructures and mechanical behaviors during processing. The Mg–Li based alloy with Sc addition presented age hardenable effect at room temperature. The hardness decreased rapidly with aging temperature at temperatures below 50°C. Thermomechanical treatment could enhance the work hardening effect to improve the mechanical properties.

Keywords: Mg-Li alloy; X-ray diffraction; Age hardening; Work hardening

1. Introduction

Magnesium is the lightest metal that can be employed in structural applications when alloyed with other elements. Research on Mg alloy focusing on mechanical properties has become very active in the last decade [1-3]. In light of hexagonal close packed (HCP) structure, Mg and its alloys have crucial drawback of poor formability, especially at room temperature, as compared with aluminum and its alloys. Since slip at room temperature is limited to the basal plane, the processing and forming capabilities of magnesium alloys are generally fairly poor.

Alloying Mg with lithium yields a lightest structural metal of Mg–Li alloy. The Mg-Li phase diagram [4] indicates that when the Li content is between ~5.5 and 11.5 wt%, the BCC structured β phase of the Li solid solution co-exists with the HCP α phase of the Mg solid solution. The β single phase structure could exist for Li contents greater than 11 wt%. As the amount of Li added to the Mg-Li alloy increases, the α phase still possesses HCP structure, but the crystal lattice axes ratio, c/a decreases such that slip between crystal planes become less difficult [5], the co-existence of the β phase makes the Mg–Li alloy possible to be cold worked. However, the mechanical properties of Mg–Li alloy are not particularly favorable for structural applications due to its low tensile and yield strength. Various third elements have been added to the Mg–Li alloy systems to explore the effect of the addition of a third element on the mechanical properties and formability [6-9].

Al addition has been selected as a precipitation and solid solution hardener [10,11], and Zn could be added to improve the cold formability [12]. Bach et al [11] demonstrated that the ductility of the Mg–Li–Al alloy could be improved by minor additions of RE elements. Scandium (Sc) has been chosen as an alloying element for improving the creep resistance of magnesium alloys [13]. Al alloys containing Sc have been developed as high strength Al alloys [14]. Minor addition of Sc results in the formation of fine dispersed Al_3Sc compound to enhance the strengthening effect in Al alloys.

Table 1 Chemical composition of the LAZ1010Sc alloy

Elements	Li	Al	Zn	Sc	Mg
wt%	10.41	1.02	0.46	0.01	Rem.

In this work, an Mg–Li–Al–Zn (designated as LAZ) alloy containing about 10wt% of Li was chosen as the matrix alloy. Main emphasis was placed on investigating the effects of minor Sc addition on the microstructures and mechanical properties of the LAZ alloy processed by heat and mechanical treatments.

2. Materials and Experimental Procedures

2.1 Alloys

The Mg–Li alloy was melted in a high-vacuum electric induction furnace under an argon atmosphere and then cast into an ingot with a cylindrical shape of 200 mm in diameter and 400 in mm height. The analyzed chemical composition of the cast alloy by use of an induction coupled plasma (ICP) and Spark Optical Emission Spectrometry (Spark-OES) apparatuses is given in Table 1. The cylindrical ingots were then extruded into a plate of 110 mm in width and 10 mm in thickness at a billet preheating temperature of 200°C.

2.2 Age heat treatment and thermomechanical processing

The as-extruded specimen for alloy LAZ1010Sc was annealed at 300°C for 1 h followed by quenching, annealed specimen was then aged at room temperature, 50, 100, 150, and 200°C for 1-16 h.

The annealed specimen was cold rolled with the reductions of 20, 40, 60, and 80%. Reference specimens for comparison were directly cold rolled from the extruded plate with the same reductions as used for the annealed specimens to explore the effect of annealing heat treatment on cold working.

2.3. Tensile Tests

Uniaxial tension test was carried out in the direction along the extrusion and rolling direction. The gauge length and width of the tensile specimen were 50 and 6 mm, respectively. The specimens were tested at room temperature with an initial strain rate of 1.67×10^{-3} s^{-1}.

2.4 Metallographic inspection

The specimen for microscopic examination was prepared by conventional metallographic techniques. The polished specimen was etched for 1~5 sec in the etchant of 5 g Picric Acid, 10 ml Acetic Acid, 95 ml Ethyl Alcohol. Optical microscopy (OM) and scanning electron microscopy (SEM) were used to examine the microstructures. X-ray diffraction (XRD) was utilized to identify the phases in the microstructures. Grain size was measured by the linear intercept method according to ASTM standard E 112-88.

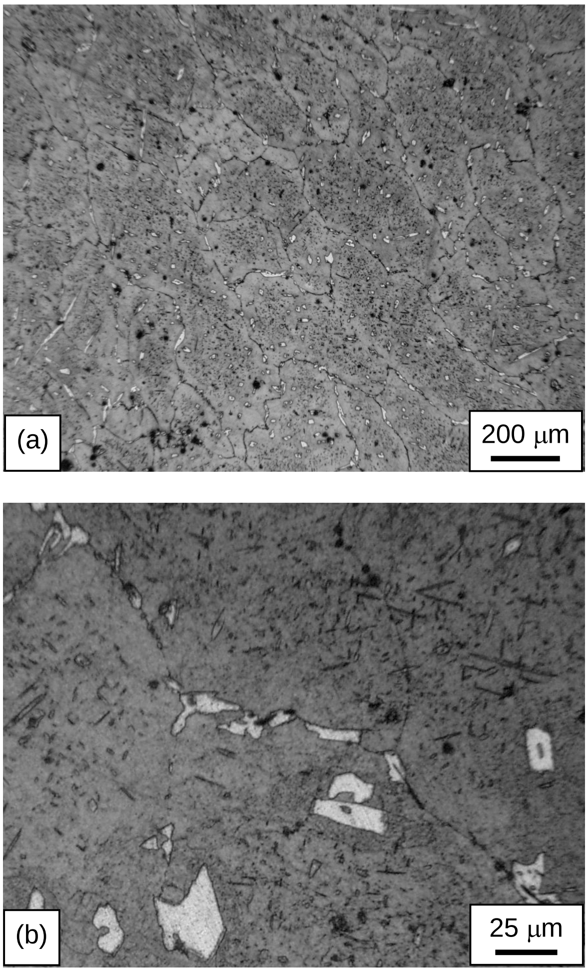


Fig.1. Optical micrographs of the as-cast microstructures. (a) LAZ1010Sc alloy, (b) LAZ1010Sc alloy on a larger scale.

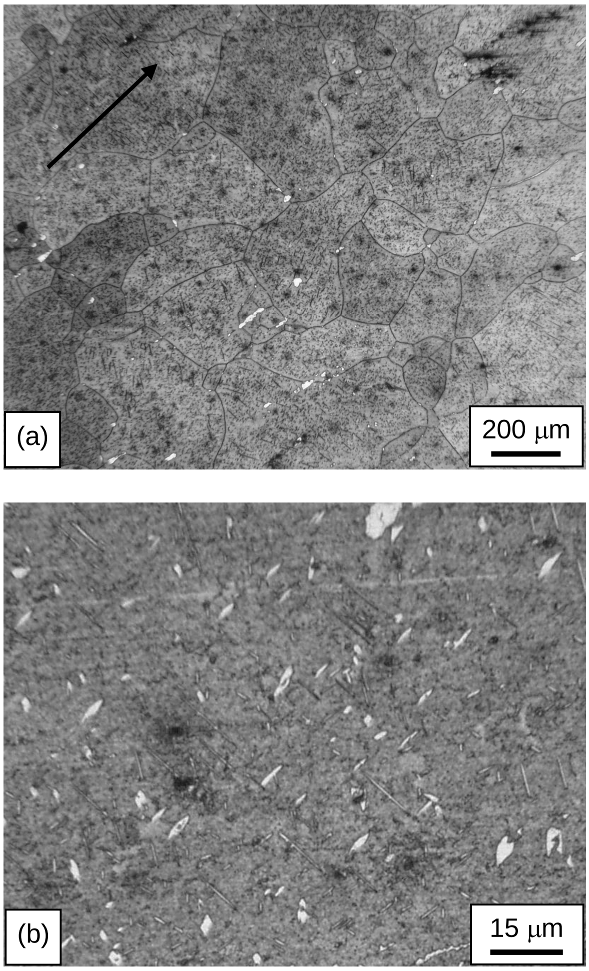


Fig.2. Optical micrographs of the as-extruded microstructures; the arrow indicating the extrusion direction. (a) LAZ1010Sc alloy, (b) LAZ1010Sc alloy on a larger scale.

3. Results and Discussion

3.1 Analysis of the microstructure

Fig. 1 shows the as-cast structures of LAZ1010Sc alloy; the cast alloys contain both the major β–Li phase and minor α–Mg phase. The Li content of LAZ1010Sc alloy is about 10.41 wt% which is near the region of single β phase, so only few dispersed α phase particles exhibit in the microstructure, as demonstrated in Fig. 1(a), and α phase exhibits two types of morphology, one is clustered particles located in the grains and along the grain boundaries, and the other is small rod-shaped particles inside the grains, as given in Fig.1(b).

Fig 2 demonstrates the as-extruded microstructure of alloy LAZ1010Sc. Fibrous structures were not observed in this study. Dynamic recrystallization should have taken place during extrusion, and some grain growth could be found in the as-extruded structure. Dynamic recrystallization of β phase was also found in the as-extruded Mg–8.7Li and Mg–8.5Li–6.4A1 alloy [15]. The rod-shaped particles still present inside the grains.

A Widmanstätten type morphology of α phase present in the β matrix was observed in the microstructure of the aged LAZ1010Sc alloy, as demonstrated in Fig. 3(a), but not found in the aged alloy of LAZ1010, as given in Fig. 3(b). The Widmanstätten structure was also found in the squeeze cast Mg–10wt%Li–3wt%Al, but not in the squeeze cast Mg–8wt%Li–3wt% Al [8].

3.2 Age heat treatment

Changes in hardness during aging for LAZ1010Sc alloy are illustrated in Fig. 4. Age hardening for LAZ1110Sc alloy could only be observed for aging at room temperature, hardness increases with aging time up to about 4~5 h, only decrease in hardness with aging time was observed for aging at temperatures higher than 50°C.

Fig. 5 shows hardness variation at various temperatures aged for 4 h. The hardness decreases rapidly with temperature at temperatures below 50°C, the rapid decrease in hardness should be related to the transformation of the θ phase into the equilibrium phase AlLi. Alloying Mg–Li with Al can form coherent metastable θ ($MgLi_2Al$) precipitate to strengthen the β phase matrix in the Mg–Li–Al alloys [16-18], however the fully hardened alloys were softened due to the transformation of θ phase into an equilibrium phase AlLi at intermediate temperatures. Fig. 5 indicates that the transformation of θ phase should be at a temperature below 50°C.

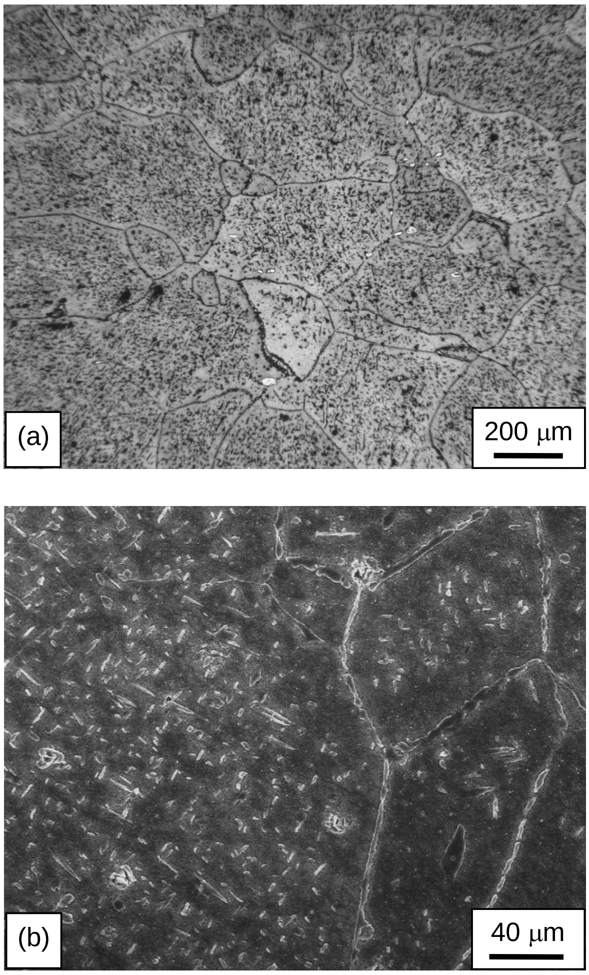
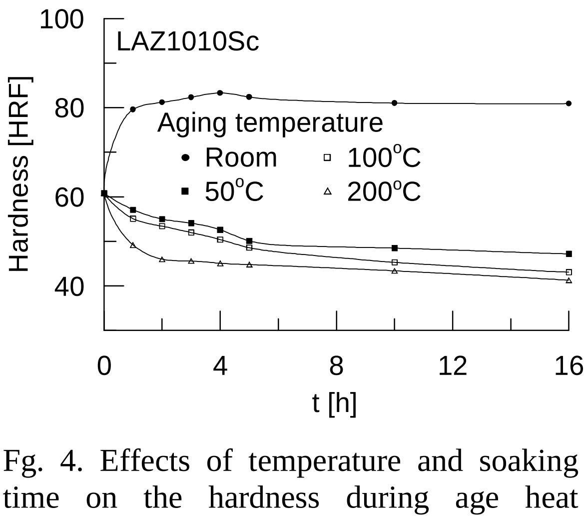


Fig.3. Microstructures of the aged specimens. (a) OM image of LAZ1010Sc alloy, (b) SEM image of LAZ1010Sc alloy on a larger scale.



Fg. 4. Effects of temperature and soaking time on the hardness during age heat treatments.

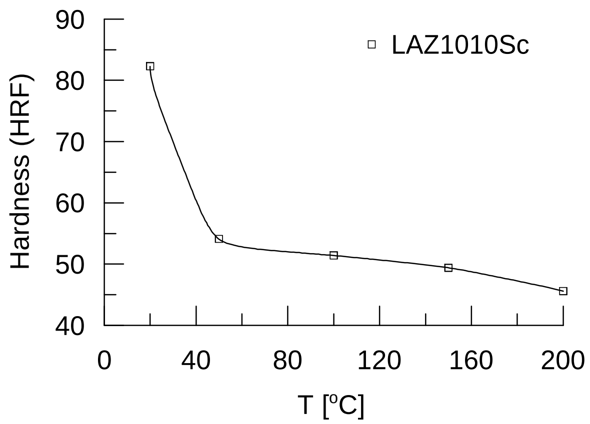


Fig. 5. Hardness variations of LAZ1010Sc alloy aged at different temperatures holding for 4 h.

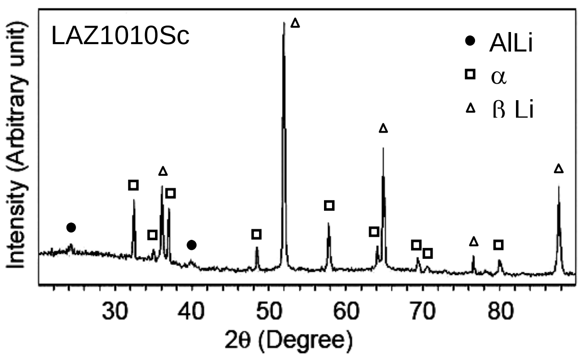


Fig. 6. Line profile analysis of XRD patterns of the LAZ1010Sc alloy aged at 150°C.

Table 2 Mechanical properties of the as-extruded and naturally aged specimens

Conditions	Yield Strength (MPa)	Tensile Strength (MPa)	Elongation (%)
As-extruded LAZ1010Sc	129.4	155.8	45.3
Aged LAZ1010Sc	172.1	186.4	27.1

The X-ray diffraction (XRD) patterns obtained from the LAZ1010Sc alloy aged at 150°C is given in Fig. 6. It shows that the LAZ1010Sc alloy comprises α–Mg, β–Li, and AlLi structures, no intermediate compounds between Al-Sc and/or Mg-Sc are presented in the LAZ1010Sc alloy with a 0.01 wt% Sc addition. Since θ phase transforms into an equilibrium phase AlLi at intermediate temperatures, only AlLi was

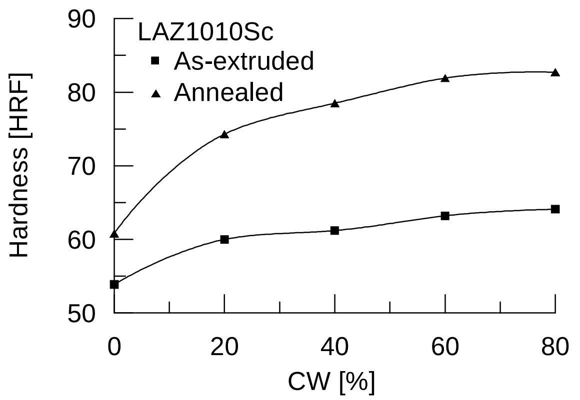


Fig. 7. Hardness variations of LAZ1010 and LAZ1010Sc alloy with different thickness reductions showing the effect of prior annealing treatment.

observed in the XRD patterns.

The values of tensile properties of the as-extruded and naturally aged specimens are listed in Table 2. The tensile strength after aging increase about 19.6%. The naturally aged LAZ1010Sc alloy gives a tensile strength of 186.4 MPa with an elongation of 27.1%.

3.3 Effect of thermomechanical processing

Fig. 7 illustrates the variation in hardness with thickness reduction for the as-extruded and annealed specimens. Increase in hardness with thickness reduction indicates that LAZ1010Sc alloy could be work hardened. For the specimens with a prior annealing treatment, the working hardening rates with reductions below 40% are higher than those of the specimens without a prior annealing treatment. Therefore, a prior annealing treatment could enhance work hardening effect for LAZ1010Sc alloy.

The mechanical properties of the specimens without a prior annealing treatment are listed in Table 3. Increases in tensile strength for a reduction of 80% is 23.2% for LAZ1010Sc alloy. Although the elongation decreases with increasing reduction, the LAZ1010Sc alloy still presents an elongation of 14.9% with a thickness reduction of 80%. Table 4 displays the mechanical properties of the specimens with a prior annealing treatment. Significant increases in strengths, compared to the properties of the as-extruded specimens, were found for the specimens with a prior annealing treatment. An increases in tensile strength for a reduction of 80% is 46.5% for LAZ1010Sc alloy.

4. Conclusions

The effects of thermal and mechanical treatments on the microstructures and mechanical behaviors of an Mg–Li–based alloy containing minor addition of Sc were investigated in this study. The as-cast structure of LAZ1010Sc alloy presented a dual phase microstructure of a β matrix and a distributed α phase, and small rod-shaped particles of α Mg phase could be observed inside the grains. The LAZ1010Sc alloy could only be naturally aged, the aged LAZ1010Sc alloy presented a Widmanstätten type morphology of α phase in the β matrix.A minor Sc addition would shift the transformation of the θ phase into the equilibrium phase AlLi to a lower temperature. The strengths of the Mg–Li based alloy could be improved by a minor addition of Sc. The naturally aged LAZ1010Sc alloy revealed a yield strength of 172.1 MPa, a tensile strength of 186.4 MPa with an elongation of 27.1%. Work hardening effect could be enhanced by a prior annealing treatment carried out on the extruded plate before cold rolling.

Acknowledgments

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Table 3 Mechanical properties of the cold rolled specimens of LAZ1010Sc alloy without a prior annealing treatment

Property	Thickness Reduction (%)				
	0	20	40	60	80
Yield Strength (MPa)	129.4	138.2	155.8	166.6	179.1
Tensile Strength (MPa)	155.8	166.6	173.5	181.4	191.9
Elongation (%)	44.7	34.2	25.7	21.2	14.9

Table 4 Mechanical properties of the cold rolled specimens of LAZ1010Sc alloy with a prior annealing treatment

Property	Thickness Reduction (%)				
	0	20	40	60	80
Yield Strength (MPa)	161.5	191.1	201.9	211.7	220.4
Tensile Strength (MPa)	184.0	207.8	218.5	223.4	228.3
Elongation (%)	22.3	17.8	14.1	12.9	10.1

計畫成果自評

本研究依據原計畫目標完成含鈧超輕鎂鋰合金之性質研究，完成之工作項目如下所述：

(1) 含鈧鎂鋰合金的熔煉
(2) 含鈧鎂鋰合金厚板擠製
(3) 含鈧鎂鋰合金精密薄板軋延
(4) 含鈧鎂鋰合金金相分析
(5) 含鈧鎂鋰合金時效硬化處理
(6) 含鈧鎂鋰合金機械性質探討
(7) 含鈧鎂鋰合金加工硬化特性探討

本計畫完成了含鈧鎂鋰合金薄板壓延製作技術及性質分析。完成碩士生研究論文「含鈧LAZ1110合金之熱處理性質研究」一篇。就研究成果之學術價值而言，適合在國外SCI等級以上之學術期刊發表。研究成果已撰寫論文一篇，投送中國材料科學學會2008年會發表。投寄於國外學術期刊之論文已撰寫完成，以論文題目”Effects of minor Scandium addition on the properties of Mg-Li-Al-Zn alloy"投寄國際期刊Journal of Alloys and Compounds，目前已被接受。

本研究計畫之主要發現，而且也未在其它學術期刊所發表之重要成果包括下列所述之幾項：

1. 在LAZ1010鎂鋰合金中添加Sc元素在微結構中出現魏德曼組織，α相會以細微顆粒及針狀結構散佈於β相中。魏德曼組織會隨著時效溫度升高而有增多之趨勢。
2. 含鈧LAZ1010合金之硬度與α相與β相之間的大小與比例含量有相當明顯之關係。原則上，α相愈小，散佈於β相中愈多，其硬度愈高。
3. 含鈧LAZ1010合金僅具有自然時效硬化的趨勢，當時效溫度升高時，硬度會隨著時間而下降的。
4. 含鈧LAZ1010合金在300°C固溶後進行室溫時效處理時，其硬度有明顯提升，50°C下時效處理則無明顯效果，100°C下時效處理時則會處於過時效狀態。含鈧LAZ1010合金在400°C固溶後進行室溫時效處理時，其硬度值有明顯提升，50°C下進行時效處理則有硬度下降的趨勢，進行100℃時效處理則導致過時效的發生。
5. LAZ1010+Sc合金具有加工硬化的效果。冷壓延前，若經退火熱處理，其硬度較未經過熱處理提升許多，但其硬度趨勢與未經過熱處理之合金類似，顯示適當之熱處理有助於提升冷加工後之硬度。