Recrystallization Behavior of CP Ti Welds by Rolling

압연에 의한 CP Ti 용접부 재결정 거동 연구

Byoung-Hyun Yoon^{*,†}, Ji-Yeon Shim^{**}, and Bong-Yong Kang^{**}

^{*}Rapidly Solidified Materials Research Center, Chungnam National Univ., Daejeon, 34134, Korea

^{**}Korea Institute of Industrial Technology, Jeonju, 54845, Korea

[†]Corresponding author: byoon007@naver.com

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Abstract

Electrolytic copper foil is a thin copper film material manufactured by electroplating and is mainly used as a circuit material to transmit electrical signals in printed circuit boards of electronic products such as TVs, PCs, and smartphones. Recently, it has been widely used as a negative current collector for lithium-ion batteries, which is essential for mobile IT and electric vehicles. The electrodeposited drum material is composed of CP Ti, which exhibits corrosion resistance and has a light weight, and welding is essential for processing in the form of a drum. Therefore, when the microstructures of the base metal and the welded part differ, these differences are reflected in the surface of the copper foil, which results in poor quality. Therefore, in this study, a welding process that required a low heat input while minimizing the size of the weld was used, and the change in the grain size was studied through plastic deformation and recrystallization heat treatment of the weld structure. Following the recrystallization heat treatment after the rolling of the plasma welded specimen, it was found that the calculation results of the recrystallization heat treatment were consistent with the actual conditions of the heat treatment, and the microstructure of the weld metal became finer as the rolling reduction rate increased.

Key Words: Electrolytic copper foil, Drum, CP Ti, Welding, Grain size, Recrystallization

1. Introduction

The electrolytic copper foil is a thin copper film fabricated through an electroplating process, which is mainly used as circuit components that transmit electrical signals in printed circuit boards of electronic products such as television, personal computer, and smartphones. Recently, their demand is increasing as the cathode current collector material, which is essentially applied in lithium-ion batteries of mobile information technology (IT) and electric vehicles. The electrolytic copper foil is fabricated with the electroplating method, using the equipment composed of a cathode (titanium), anode (titanium), and busbar (copper). This process consists of creating a copper sulfate solution for fabricating the copper foil by melting highly pure cop-

per into a sulfuric acid solution, electroplating the copper ions from the copper sulfate solution to a large-scale Ti drum (cathode) in the electrolyzer, and exfoliating to create a rolled product. The commercially pure (CP) Ti is used as an electroplating drum material by considering the corrosion resistance, light weight, and detachability where processing it into a drum shape requires welding. However, a microstructural variation in the base material and the weld zone results in a defective product due to the transcription to the copper foil surface. Thus, microstructural adjustment is necessary to have a weld zone structure similar to the base material structure. Kang¹⁾ reported that the soldering process of the CP Ti material during the friction stir welding had been achieved at a β -transformation temperature or below and no phase transformation occurred in the CP Ti, while phase transformation oc-

curred in the Ti-6Al-4V alloy because the processing temperature reached the β -transformation temperature or above. According to Moon²⁾, the MC (M: V and/or Nb) carbide was extracted at the transgranular zone and crystal grain boundary in the welding of the austenitic Fe-30Mn-9Al-0.9C (wt%), in which Nb and V were added, and crystal grain boundary growth inhibition effect was observed at the heat-affected zone. Tan³⁾ performed hot rolling of the CP Ti material in the 600 - 800 °C range and air cooling thereafter. Consequently, the recrystallization behavior of the crystal grain was observed. Hayashi et al.⁴⁾ also performed hot rolling of CP Ti at 600 - 800 °C according to the varying reduction rate, which showed variations in rolled structure such as the generation of a twin crystal due to the forming process generated during rolling. Hayashi et al.⁴⁾ reported that the structure became finer due to recrystallization during the cooling process and that the structure transformed due to static recrystallization according to the temperature and reduction rate. In this study, a low heat input welding process, which could minimize the weld zone size, was utilized. Furthermore, the variation in grain size was studied through the forming process of the weld zone structure and recrystallization annealing

2. Materials and Experimental Methods

2.1 Materials

The material used in this study was a 9-mm grade 1 CP Ti, and the analysis results of its chemical substances and mechanical properties are summarized in Table 1. As a grade 1 material containing 0.025 wt% Fe, the elongation rate was 39% and tensile strength 324 MPa.

Fig. 1 presents an image of the microstructure of the material used, in which grain size measurement results revealed that their degree was at level No.9.9 of the American Society for Testing and Materials (ASTM).

2.2 Welding process

The welding of the Ti material proceeded in two parts.

Table 1 The materials used in this study (wt%)

Fe	C	N	O	H	Ti
0.025	0.0185	0.0042	0.16	0.0103	Bal
Yield strength (N/mm ²)		Tensile strength (N/mm ²)		Elongation (%)	
238		324		39

Fig. 1 Photograph showing the microstructure of the base metal

Fig. 2 Schematic diagram of the welding process

Additional lamination was required to the base material thickness of 9 mm for the weld zone rolling. The weld zone specimen was fabricated so that the entire height becomes 19 mm through plasma welding and weld bead deposition. First, plasma keyhole welding was used to minimize the heat effect at the weld zone. After the welding, the weld bead was deposited to be applied in the rolling process.

The plasma keyhole welding was performed with the I-type and without root gaps. The welding current was approximately 275 A, the welding speed was 30 cm/min, and the heat input was around 18 kJ/cm. Here, Ar gas was additionally supplied at 25 L/min to protect the entire weld zone from the atmosphere.

The plasma process and the CP Ti grade 1 welding rod were used to perform the build-up welding. Likewise,

Fig. 3 Schematic diagram of rolling process used in this study

Ar gas was supplied at 12 L/min during welding to protect the weld zone.

2.3 Weld zone rolling

The specimen that was fabricated under the same conditions mentioned above was used in the rolling process shown in Fig. 3. The reduction rate was adjusted to be 80% in total at room temperature for the rolling. Here, the rolling reduction per rolling frequency was 1 - 2 mm, and the total rolling frequency according to the reduction rate was 20 - 30 times.

2.4 Structural analysis and grain size measurement

The junction was mechanically polished for analyzing the weld zone structure and measuring the grain size. Then, it was etched at room temperature for five seconds with a mixed solution of 100 ml distilled water + 3 ml hydrofluoric acid + 4 ml nitric acid. A deformed structure forms in the CP Ti material even during mechanical polishing. Thus, mechanical polishing and etching were executed multiple times to remove these structures. In the microstructural observation, an optical microscope was utilized to observe the macroscopic structural transformation, and the grain size was measured according to the ASTM method at 100x magnification.

3. Experimental Results and Discussion

3.1 Weld zone structure

The specimen fabricated by plasma welding and build-up welding using this are shown in Fig. 4. As mentioned above, the height of one weld bead was 8 mm, and the opposite side had a 2 mm weld bead layer.

The cross-section of the welded specimen is as provided in Fig. 5 in which twin crystals were partially found at the ferrite matrix structure in the deposited metal.

Fig. 4 Welded specimen

Fig. 5 Optical microstructures of specimen as welded (a) low magnification, (b) weld metal at build-up welding, (c) weld metal at keyhole welding, and (d) heat affected zone

3.2 Results of rolling

The welded specimen was cut to approximately 30 mm in width and 50 mm in length to perform rolling. On the side with the high weld bead layer, cracks were observed at the surface as the reduction rate increased, and the cracks were more frequent as the reduction rate increased. On the opposite side, the weld zone and the base material were simultaneously rolled as shown in Fig. 6.

3.3 Recrystallization temperature and time calculation

The Avrami equation was used to calculate the recrystallization temperature and time for refining the grain size of the rolled specimens through recrystallization annealing. The Avrami equation is shown in Equation (1) where the time at 50% transformation is represented as a variable. This equation was used to calculate the recrystallization temperature and time. For the calcu-

Fig. 6 Specimens after rolling, (a) reduction rate 20%, (b) reduction rate 50%, and (c) reduction rate 80%

Table 2 $t_{0.5}$ values and avrami constants at varying annealing temperatures⁵⁾

Annealing temperature (°C)	500	600	800
n	1.0	1.2	1.0
$t_{0.5}(\text{min})$	6060	50	0.2

lation, the data presented in Table 2 were utilized for the Avrami exponent (n) and $t_{0.5}$ ³⁾.

$$X = 1 - \exp \left\{ -B \left(\frac{t}{t_{0.5}} \right)^n \right\} \quad (1)$$

X: Area fraction of recrystallized grains

t: Annealing time

$t_{0.5}$: Time at 50% recrystallization

n : Avrami exponent

B: Constant, CP Ti=0.693

As shown in Table 2, the Avrami exponent n was reported to vary according to the annealing temperature. At $n = 1$, the annealing time increased significantly. Therefore, the evaluation was proceeded with the lowest annealing time possible considering the circumstances of actual annealing.

Fig. 7 presents the calculation results in a graph when the Avrami exponent n was 1.2 and the recrystallization temperature was 500 - 800 °C. At 500 °C, the duration of recrystallization until completion was 46,500 minutes, which was excessively long for the actual application. At 600 °C, 650 °C, and 700 °C or higher, this time was reduced to 280 minutes, 80 minutes, and within about 10 minutes, respectively.

3.4 Recrystallization annealing results

In Fig. 8, which presents the microstructure images of

Fig. 7 Calculation results of recrystallized fraction and time at varying annealing temperatures **Fig. 8** Optical microstructure of welded specimens after being rolled and annealed at 650 °C, (a) reduction rate 20%, (b) reduction rate 50%, and (c) reduction rate 80%

the rolled specimens at reduction rates of 20%, 50%, and 80% after annealing at 650 °C for 80 minutes, recrystallization was observed in all the specimens. In addition, the deposited metal and the heat-affected zone were refined as the reduction rate increased. In the specimen with a reduction rate of 80%, the microstructures between the base material and weld zone were indistinguishable.

The measurement results of the rolled and annealed specimen grain size showed that the grain size became finer as the reduction rate increased, as shown in Fig. 9

Fig. 9 Measurement results of grain size against reduction rate

Fig. 10 Optical microstructures of the specimen as heat treated: (a) reduction rate 20% at weld metal, (b) reduction rate 50% at weld metal, (c) reduction rate 80% at weld metal, (d) reduction rate 20% at HAZ, (e) reduction rate 50% at HAZ, and (f) reduction rate 80% at HAZ

and 10. When the reduction rate was 20%, the grain size of the deposited metal was about 7.3 ASTM, but it was 9.7 after the reduction rate increased to 80%, which was a similar grain size to the base material. This was considered as the amount of transformation for recrystallization increasing as the reduction rate increased and the increased accumulated energy contributing to the grain size refining.

Meanwhile, the grain sizes at the heat-affected zone and the deposited metal vary, which was considered to be relevant to the laminated bead layer form. As shown in Fig. 8, the rolling force was concentrated towards the downward direction at the thick center of the weld zone bead layer. This was considered to have occurred from no rolling force being directly delivered to the heat-affected zone due to the morphological properties of the weld zone. Therefore, it was determined that the weld bead layer form must be considered to reduce the microstructural variation between the deposited metal and the heat-affected zone.

4. Conclusions

The following results were drawn by rolling and recrystallization annealing the weld zone of the Ti cathode drum for electrolytic copper foil fabrication.

1) A specimen was fabricated by plasma welding the CP Ti material. A specimen that formed 8 mm and 2 mm weld bead layers at the top and bottom, respectively, was fabricated for the rolling.

2) The results of rolling the welded specimens showed that cracks were generated at the side where the weld bead layer height was 8 mm, while cracks were absent at the side with the weld bead layer height of 2 mm when rolling until 80%.

3) The Avrami equation was used to calculate the recrystallization temperature and time and determine the recrystallization condition. Using the results, the annealing experiment was proceeded at 650 °C for 80 minutes after which recrystallization was completed. As the reduction rate increased, a decrease in the grain size variation between the deposited metal and base material was observed.

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ORCID: Byoung-Hyun Yoon: <http://orcid.org/0000-0003-4683-7888>

ORCID: Ji-Yeon Shim: <https://orcid.org/0000-0002-2825-9423>

ORCID: Bong-Yong Kang: <https://orcid.org/0000-0002-7826-9873>

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Recrystallization Behavior of CP Ti Welds by Rolling

윤 병 현* · 심 지 연** · 강 봉 용**

*충남대학교 급속응고신소재연구소

**한국생산기술연구원 전북지역본부

1. 서 론

전해동박은 전기도금 공정으로 제조되는 얇은 구리 박막소재로 주로 TV, PC, 스마트폰 등 전자제품의 인쇄회로기판에서 전기신호를 전달하는 회로소재로 사용이 된다. 최근에는 모바일 IT, 전기자동차 등에서 필수적으로 적용되는 리튬이온전지의 음극집전체 소재로도 수요가 확대되고 있는 소재이다. 이러한 전해 동박은 음극(티타늄), 양극(티타늄) 및 부스 바(구리)로 구성된 제조설비를 이용한 전기도금법으로 제조하는데, 그 공정은 고순도의 구리(Cu)를 황산용액에 녹여 동박 제조를 위한 전해액(황산구리용액)을 만들고 전해조에서 황산구리용액으로부터 구리 이온을 대형 Ti Drum(음극전극)에 전착시킨 후 박리하여 Roll제품으로 생산한다.

전착드럼 소재는 내부식성, 경량성 및 박리성 등을 고려하여 CP Ti 소재를 사용하는데, 드럼 형태로 가공하기 위해서는 용접이 필수적이다. 따라서 모재와 용접부의 미세조직적 차이가 발생하게 되면 동박 표면에 전사되어 품질불량을 초래하므로 용접부의 조직이 모재조직과 유사하도록 조직 제어가 필요하다.

Kang¹⁾ 등에 의하면 CP Ti 소재의 마찰교반접합시 접합공정이 β -변태온도 이하에서 이루어지기 때문에 달성되었기 때문에 CP Ti에서는 상 변형이 발생하지 않았다고 보고하고 있으며, Ti-6Al-4V 합금에서는 공정 온도가 β -변태 온도 이상에 도달하여 상변태가 발생한다고 보고하고 있다. Moon²⁾ 등에 의하면 Nb, V를 첨가한 오스테나이트계 Fe-30Mn-9Al-0.9C(wt%) 경량철강의 용접에서 MC(M: V and/or Nb) 탄화물이 결정립계 및 입내에 석출되어 열영향부에서 결정립 성장 억제 효과가 있었다고 보고하고 있다.

Tan³⁾ 등은 CP Ti 소재를 600℃ ~ 800℃ 범위에서 열간압연을 실시한 후 공냉을 한 결과, 결정립이 미세화되는 재결정 거동을 관찰한 바 있으며, Hayashi⁴⁾ 등도

600℃ ~ 800℃ 범위에서 CP Ti 소재를 압하율 변화에 따라 열간압연을 실시한 결과, 압연시 발생한 소성가공의 영향으로 쌍정이 형성되는 등의 압연조직의 변화가 보이고 생각하는 과정에서 재결정되어 조직이 미세해진다고 보고하고 있으며, 온도 및 압하율에 따라 정적 재결정현상으로 조직이 변화하였다고 보고하고 있다.

본 연구에서는 용접부의 크기를 최소화 할 수 있는 저입열 용접공정을 이용하고 용접부 조직을 소성가공 및 재결정 열처리를 통하여 결정립도 변화를 연구하고자 하였다.

2. 사용 재료 및 실험 방법

2.1 사용 재료

본 실험에 사용된 소재는 두께 9mm의 CP Ti grade 1 소재로 화학적 성분 및 기계적 성질을 분석한 결과를 Table 1에 나타내었다. Fe가 0.025wt% 함유되어 있는 Gr.1 소재로 인장강도는 324MPa, 연신율은 39% 이었다.

Fig. 1은 본 소재의 미세조직을 보여주는 사진으로 결정립도 측정 결과 ASTM No. 9.9 정도인 것으로 분석되었다.

2.2 용접 공정

Ti 소재의 용접은 두 부분으로 나누어 진행하였다.

Table 1 The materials used in this study

(wt%)

Fe	C	N	O	H	Ti
0.025	0.0185	0.0042	0.16	0.0103	Bal
Yield strength (N/mm ²)		Tensile strength (N/mm ²)		Elongation (%)	
238		324		39

Fig. 1 Photograph showing the microstructure of the base metal

Fig. 2 Schematic diagram of the welding process

용접부 압연을 위해서는 모재 두께 9mm이외에 추가적인 적층이 필요하였으며 플라즈마 용접과 용접 비드 적층을 통해 전체 높이를 19mm가 되게 용접부 시편을 제작하였다. 우선 용접부의 열영향을 최소화하기 위해 플라즈마 키홀 용접을 이용하여 용접한후 그 위쪽에는 용접비드를 적층하여 압연공정에 적용하고자 하였다.

플라즈마 키홀 용접은 I형으로 root gap없이 용접하였으며 용접전류는 약 275A, 용접속도는 30cm/min로 입열량은 약 18kJ/cm이었다. 이때 용접부 전체를 대기로 부터 보호하기 위해 Ar가스를 25L/min의 양으로 추가 공급하였다.

빌드업 용접은 역시 플라즈마공정을 이용하여 CP Ti grade 1 용접봉을 이용하여 적층용접을 실시하였다. 마

Fig. 3 Schematic diagram of rolling process used in this study

찬가지로 용접부 보호를 위해 Ar가스를 약 12L/min의 양으로 공급하면서 용접을 실시하였다.

2.3 용접부 압연

상기와 같은 조건으로 제작된 시편을 이용하여 Fig. 3과 같은 압연공정을 이용하여 압연을 실시하였다. 압연은 상온에서 압하율이 총 80%가 되도록 하였으며, 이때 압연 횟수당 압하량 1~2mm로 압하율에 따라 총 압연 횟수는 20~30 회 이었다.

2.4 조직 분석 및 결정립도 측정

용접부 조직 분석 및 결정립도 측정을 위하여 접합부를 기계적으로 연마한 후 증류수 100ml + 불산 3ml + 질산 4ml의 혼합용액으로 약 5초간 상온에서 에칭하였다. CP Ti 소재는 기계적 연마중에도 가공조직이 만들어지기 때문에 이러한 조직의 제거를 위하여 기계적 연마와 에칭을 수회 실시하였다. 미세조직 관찰은 광학현미경으로 거시적 조직 변화를 관찰하였고, 결정립도 측정은 ASTM 방법에 의하여 100배 배율에서 측정하였다.

3. 실험 결과 및 고찰

3.1 용접부 조직

플라즈마 용접과 이를 이용한 적층용접으로 제작된 시편은 Fig. 4와 같으며 전술한 바와 같이 한쪽의 용접비드 높이는 8 mm, 반대쪽 면은 2mm의 용접비드 층을 갖는 형상이다.

용접시편의 단면 형상은 Fig. 5에 나타낸 바와 같으며, 용착금속에서는 페라이트 기지조직에 일부 쌍정이 발견되었다.

Fig. 4 Welded specimen

Fig. 5 Optical microstructures of specimen as welded (a) low magnification, (b) weld metal at build-up welding, (c) weld metal at keyhole welding, and (d) heat affected zone

3.2 압연 결과

용접된 시편을 약 폭30mm, 길이 50mm 정도로 절단하여 압연을 실시하였다. Fig. 6은 압연 후 시편을 보여주는 사진으로 용접 비드층이 높은 면에서는 압하율이 증가할수록 표면에 균열들이 보였으며, 압하율이 커질수록 균열은 더 많이 나타났다. 반대 면에서는 용접부와 모재가 동시에 압연되는 형상을 보여주고 있다.

3.3 재결정 온도 및 시간 계산

압연이 완료된 시편을 재결정 열처리를 통하여 결정립을 미세화하기 위해서 Avrami 방정식을 이용하여 재결정 온도와 시간을 계산하였다. 식(1)은 변태가 50% 완료되는 시간을 변수로 나타낸 Avrami 방정식으로 이 식을 이용하여 재결정 온도 및 시간을 계산하였다. 계산을 위한 Avrami 지수(n)과 $t_{0.5}$ 는 Table 2에 나타낸 바와 같은 자료를 활용하였다³⁾.

$$X = 1 - \exp \left\{ -B \left(\frac{t}{t_{0.5}} \right)^n \right\} \quad (1)$$

Table 2 $t_{0.5}$ values and Avrami constants at varying annealing temperatures⁵⁾

Annealing temperature (°C)	500	600	800
n	1.0	1.2	1.0
$t_{0.5}(\text{min})$	6060	50	0.2

Fig. 6 Specimens after rolling: (a) reduction rate 20%, (b) reduction rate 50%, and (c) reduction rate 80%

X : Area fraction of recrystallized grains

t : Annealing time

$t_{0.5}$: Time to 50% recrystallization

n : Avrami exponent

B : Constant, CP Ti=0.693

Table 2에 나와 있듯이 Avrami 지수, n 은 온도에 따라 다른 것으로 보고되고 있는데, $n=1$ 로 계산을 하면 열처리 시간이 증가하기 때문에 실제 열처리 상황을 고려하여 가능한 열처리 시간이 짧은 쪽으로 평가를 하고자 하였다.

Fig. 7은 Avrami 지수, $n=1.2$ 로 하여 재결정 온도 500°C ~ 800°C 범위일 때 계산 결과를 보여주는 그래프로 500°C에서 재결정이 완료되는 시간은 약 46,500분으로 장시간이 소요되어 실제로는 사용할 수 없는 조건이었으며, 온도가 600°C 일 때 280분, 650°C 일 때는 80분으로 줄어드는 경향이 있고 700°C 이상의 고온에서는 약 10분 이내로 줄어든다.

3.4 재결정 열처리 결과

Fig. 8은 압하율이 각각 20%, 50%, 80%인 압연된 시편을 650°C에서 80분간 열처리 실험후 마크로

Fig. 7 Calculation results of recrystallized fraction and time at varying annealing temperatures

Fig. 8 Optical microstructure of welded specimens after being rolled and annealed at 650°C: (a) reduction rate 20%, (b) reduction rate 50%, and (c) reduction rate 80%

조직사진으로 시편 모두에서 재결정이 일어났음을 확인할 수 있었으며 압하율이 증가할수록 용착금속과 열영향부의 조직이 미세화 되는 것을 알 수 있으며 압하율이 80% 인 시편에서는 모재와 용접부와 구별이 되지 않을 정도로 유사한 미세조직을 가짐을 확인할 수 있었다.

압연 및 재결정 열처리된 시편의 결정립을 측정된 결과 Fig. 9 와 10에서 보는 것처럼 압하율이 증가하면서 결정립도 함께 미세해 지는 것을 알 수 있다. 즉, 압하율 20% 일 때 용착금속의 결정립크기는 약 ASTM

Fig. 9 Measurement results of grain size against reduction rate

Fig. 10 Optical microstructures of the specimen as heat treated: (a) reduction rate 20% at weld metal, (b) reduction rate 50% at weld metal, (c) reduction rate 80% at weld metal, (d) reduction rate 20% at HAZ, (e) reduction rate 50% at HAZ, and (f) reduction rate 80% at HAZ

번호로 7.3정도 였으나 압하율이 80%로 증가한 경우에는 ASTM 번호 9.7 로 모재와 유사한 결정립 크기를 나타내었다. 이는 압하율이 증가하면서 재결정을 위한 소성변형량이 증가하고 이렇게 축적된 에너지가 증가하여 결정립 미세화에 기여한 것으로 판단된다.

한편 용접열영향부와 용착금속에서의 결정립크기는 차이가 발생하는 데 이는 적층된 비드층의 형상과 관련이 있는 것으로 판단된다. Fig. 8에서처럼 용접부 비드층의 두꺼운 중앙부에서는 압하력이 아래 방향으로 집중적으로 전달이 되는데 용접부 형상의 특성상 용접열영향부에서는 직접적으로 압하력이 전달되지 않아 나타나는 현상으로 보인다. 따라서 용착금속과 용접열영향부의 미세조직 차이는 줄이기 위해서는 용접 비드층의 형상도 고려해야 할 것으로 판단된다.

4. 결 론

전해동박 제조용 Ti 음극드럼의 용접부에 대한 압연 및 재결정열처리를 통하여 다음과 같은 결론을 도출하였다.

1) CP Ti 소재를 플라즈마 용접으로 시편을 제작하고 압연을 위하여 상하면에 각각 8mm, 2mm 되는 용접비드층을 형성한 시편을 제작하였다.

2) 용접시편의 압연결과, 총 80%까지 압연시 압연이 진행되면서 용접비드층 높이가 8mm인면에서는 균열이

발생하였으나, 용접비드층 높이가 2mm인 면에서는 균열이 발생하지 않았다.

3) 재결정 조건을 계산하기 위하여 Avrami 방정식을 이용하여 재결정 온도 및 시간을 계산하였으며, 그 결과를 활용한 650℃에서 80분 열처리 실험한 결과 재결정이 완성된 것을 확인할 수 있었으며, 압하율이 증가할수록 용착금속과 모재와의 결정립크기 차이가 줄어드는 것을 확인하였다.

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ORCID: Byoung-Hyun Yoon: <http://orcid.org/0000-0003-4683-7888>

ORCID: Ji-Yeon Shim: <https://orcid.org/0000-0002-2825-9423>

ORCID: Bong-Yong Kang: <https://orcid.org/0000-0002-7826-9873>

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