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Examining The Phase Formation of Aging and Shallow Cryogenic Process Applied to Aluminum Alloys with Thermal Analysis

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Anahtar Kelimeler

7075 Al alaşımı
Termal analiz
Kriyojenik İşlem
Retrogresyon ve yeniden yaşlandırma

Graphical/Tabular Abstract (Grafik Özet)

In this study, retrogression and re-aging heat treatments were combined with cryogenic processes and the transformations of the phases formed were examined by thermal analysis./ Bu çalışma ile retrogresyon ve yeniden yaşlandırma ısıl işlemleri kriyojenik işlem ile kombinasyonları yapılarak oluşan fazların dönüşümleri termal analizler ile incelenmiştir.

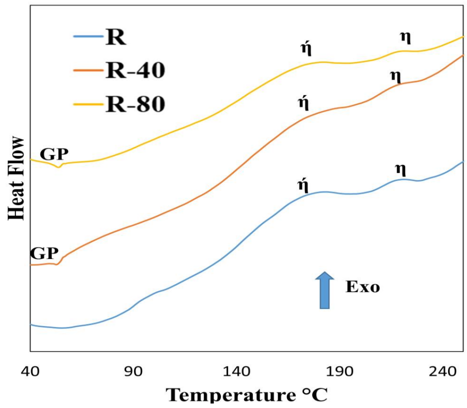

***Figure A**: Phase transformations with thermal analysis /**Şekil A:** Termal analizler ile faz dönüşümleri*

Highlights (Önemli noktalar)

- *Cryogenic combinations with retrogression and re-aging/ Retrogresyon ve yeniden yaşlandırma ile kriyojenik kombinasyonlar*
- *Examination of changing phase transformation temperatures with thermal analysis/ değişen faz dönüşüm sıcaklıkların termal analizler ile incelenmesi*
- *Activation energy calculations of the formed phases using different methods/oluşan fazların farklı yöntemlerle aktivasyon enerjisi hesaplamaları*

***Aim (Amaç):** Examination of phase transformations that occur with aging and cryogenic process with thermal analysis. / yaşlandırma ve kriyojenik işlem ile oluşan faz dönüşümlerinin termal analizler ile incelenmesi*

***Originality (Özgünlük):** Failure to investigate the kinetics of the phases formed by combining the retrogression and re-aging process with the cryogenic process. / daha önce Retrogresyon ve yeniden yaşlandırma işlemi kriyojenik işlem ile birleştirilerek oluşan fazların kinetiklerinin araştırılmaması*

***Results (Bulgular):** With cryogenic treatment, the activation energy, which was 200 kJ/mol, decreased to 100 kJ/mol. / Kriyojenik işlem ile 200 kJ/mol olan aktivasyon enerjisi 100 kJ/mol değerlerine kadar düşmüştür*

***Conclusion (Sonuç):** In the DSC graphs, it was observed that GP transformations occurred clearly in the samples subjected to cryogenic treatment. Additionally, it has been observed that activation energy decreases when cryogenic treatment is applied. This decrease shows that the ή phase will form more easily through the diffusion mechanism./DSC grafiklerinde kriyojenik işleme tabi tutulan örneklerde GP dönüşümlerinin net bir şekilde meydana geldiği gözlendi. Ayrıca kriyojenik arıtma uygulandığında aktivasyon enerjisinin azaldığı gözlenmiştir. Bu azalma difüzyon mekanizmasıyla ή fazının daha kolay oluşacağını göstermektedir.*

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Abstract

In this study, cryogenic treatment was applied to the 7*** series alloy, which is one of the aluminum alloys frequently used in the aviation and space industry, after the retrogression and re-aging process, and the phase formations were examined by thermal analysis. First of all, solution heat treatment was applied at 480 °C for 2 hours and water was given. After quenching, artificial aging heat treatment was applied at 120 °C for 24 hours. To start the RRA (retrogression and re-aging) heat treatment, after artificial aging, retrogression was performed at 200 °C for 10 minutes and quenched. Then, re-aging was performed at 120 °C for 24 hours and the aging process was completed. After the RRA heat treatment, cryogenic treatment was applied for 2 hours at -40 °C, -80 °C respectively. The heat treated samples were analyzed with a differential thermal analyzer and the transformations of GP, η′ and η phases were found. Since the η′ phase is known as the strength-increasing phase in the structure, the activation energies of each sample were calculated using the Augis-Bennet and Kissinger equations. The results showed that the activation energy of the sample treated with -40 cryogenic treatment was 50% less than the sample without cryogenic treatment. This situation proved with the Arrhenius equation that the formation of the η′ phase would be easier.

Alüminyum Alaşımlarına Uygulanan Yaşlanma ve Sığ Kriyojenik İşlemin Faz Oluşumunun Termal Analizle İncelenmesi

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Öz

Bu çalışmada havacılık ve uzay endüstrisinde sıklıkla kullanılan alüminyum alaşımlarından biri olan 7*** serisi alaşımına retrogresyon ve yeniden yaşlandırma işlemleri sonrasında kriyojenik işlem uygulanmış ve termal yöntemlerle faz oluşumları incelenmiştir. Öncelikle 480 °C'de 2 saat çözelti ısıl işlemi uygulandı ve su verildi. Söndürme işleminin ardından 120°C'de 24 saat yapay yaşlandırma ısıl işlemi uygulandı. RRA (retrogresyon ve yeniden yaşlandırma) ısıl işlemine başlamak için yapay yaşlandırmanın ardından 200 °C'de 10 dakika süreyle retrogresyon uygulandı ve söndürüldü. Daha sonra 120 °C'de 24 saat süreyle yeniden yaşlandırma yapılarak yaşlandırma işlemi tamamlandı. RRA ısıl işleminin ardından sırasıyla -40 °C ve -80 °C'de 2 saat kriyojenik işlem uygulandı. Isıl işlem görmüş numuneler diferansiyel termal analiz cihazı ile analiz edilmiş ve GP, η' ve η fazlarının dönüşümleri bulunmuştur. η' fazı yapıda mukavemet arttırıcı faz olarak bilindiğinden her numunenin η' fazının aktivasyon enerjileri Augis-Bennet ve Kissinger denklemleri kullanılarak hesaplandı. Sonuçlar, -40°C kriyojenik işleme tabi tutulan numunenin aktivasyon enerjisinin, kriyojenik işlem uygulanmayan numuneye göre %50 daha az olduğunu gösterdi. Bu durum Arrhenius denklemi ile η' fazının oluşumunun daha kolay olacağını kanıtladı.

1. INTRODUCTION (GİRİŞ)

Al-Zn-Mg-Cu (7xxx) alloys, preferred in the aviation and space industry, are an aluminum alloy group that can be reached to the highest strength levels with various aging heat treatment techniques. These alloys, which normally have high ductility, naturally have good forming properties [1-3]. However, due to the effect of aging heat treatment, their formability decreases very quickly and continues to change as the aging time increases. In general, the aging process of ageable aluminum

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alloys begins with solution heat treatment between 550-400℃ and natural aging at room temperature after quenching. The purpose of the aging heat treatment is to ensure the formation of a new phase from a supersaturated solid phase under the influence of time and temperature [4-7]. It was discovered by Cina in the 1970s that the heat treatment known as Retrogression and Re-Aging (RRA), that is, re-solution and re-aging, increases both the strength and corrosion crack resistance in 7xxx series aluminum alloys. RRA heat treatment begins with the short-term re-solution and quenching of the alloy, which has been subjected to T6 or T651 heat treatment, at a temperature range of 160-280 °C. It then continues with the re-aging process, as in the T6 artificial aging heat treatment. With RRA heat treatment, T6 and T73 properties are achieved together. Dimensional changes of the precipitates formed in the RRA process have significant effects on the yield strength of the alloy [8]. In order to understand which microstructural parameters control the corrosion susceptibility of this alloy, studies have been conducted to characterize microstructural differences under various heat treatment conditions. It has been observed that the microstructure of Al 7075-T73 shows much coarser grain boundary precipitates than those in the T6 temper. This combination has been found to result in good performance in terms of both resistance to stress corrosion cracking and mechanical strength [9-11]. In the early stages of the RRA heat treatment, a decrease in yield strength occurs due to the partial dissolution of the GP regions. As ή phases form and reach a stable size, the yield strength increases over time. It has been shown in different studies that there is a decrease again due to the coarsening of the precipitates over time. It has been shown in TEM studies that during the dissolution of GP regions, the growth of the pre-existing ή phase and the formation of new ή precipitates on the undissolved GP regions can occur simultaneously [12]. With the dissolution of GP regions, the matrix becomes enriched in zinc and magnesium. This allows the nucleation and growth of the ή phase. It has been found that this dissolution is more effective at temperatures above 180°C. As the retrogression time increases, the volume fractions and sizes of the precipitates also increase. Therefore, dissolution time is of mechanical importance. Increasing the retrogression period causes especially the η precipitate to become coarser. Since ή precipitates are less than the critical size and are unstable at this phase retrogression temperature, they will begin to dissolve slowly when the first heat treatment begins [11-13]. In the re-aging heat treatment, the mechanism of growth and re-precipitation of partially dissolved ή precipitates is active. In some studies, it has been said that the RRA structure contains more nucleated precipitates than in the T6 heat treatment condition. The higher hardness as a result of RRA heat treatment compared to the T65 heat treatment condition is explained by the presence of more and finer ή precipitates [14]. In the re-aging heat treatment, the mechanism of growth and re-precipitation of partially dissolved ή precipitates is active. In some studies, it has been said that the RRA structure contains more nucleated precipitates than in the T6 heat treatment condition. The higher hardness as a result of RRA heat treatment compared to the T65 heat treatment condition is explained by the presence of more and finer ή precipitates. It has been argued that GP regions may also grow during re-aging, but the nucleation and growth of the ή phase is the effective mechanism in re-aging [13,14].

They said that thermal analysis is one of the most used techniques to analyse the behavior of aluminum alloys in order to analyse the precipitation of Guinier-Preston (GP) zones and the different phases formed. They showed that it is possible to calculate the activation energies of the resulting phases using DSC [15].

Natural aging, artificial aging and RRA have been studied in many studies in the literature. The corrosion resistance, mechanical properties and phase transformations of these heat treatments were examined with thermal analysis. However, the changes in the transformation temperatures and activation energies of the phases formed by combining these thermal processes with cryogenic processes have not been studied before. This study, for the first time in the literature, provided a different perspective on phase transformations in aluminum alloys and enabled detailed calculations in terms of physical metallurgy. With the applied cryogenic process, more formation of the ή phase was achieved, and this was supported by thermal analyses.

2. MATERIALS AND METHODS (MATERYAL VE METOD)

In the experimental study, commercially purchased 7***series aluminum alloy was used. Solution heat treatment was applied in a high-temperature furnace at 480 °C for 2 h and then quenched. The aging process was heated to 120 °C in a horizontal high temperature furnace and an artificial aging heat treatment was applied for 24 hours. To begin the RRA heat treatment, artificial aging was followed by retrogression at 200 °C for 10 min and quenched. The aging process was then completed by re-aging at 120 °C for 24 hours. The heat treatments were carried out in an argon atmosphere. These samples are coded R. Following the RRA heat treatment, cryogenic treatment was applied for 2 hours at -40 °C, -80 °C, respectively, in an HST DWC branded cooling device, and the samples were coded as R-40 and R-80. Phase transformation temperatures and activation energies of the samples were determined by DSC analysis with the HİTACHİ DSC 7020 brand device. Tests were carried out in the temperature range of 40°C to 250°C, with four different heating rates. Figure 1 shows the flow chart of the experimental studies.

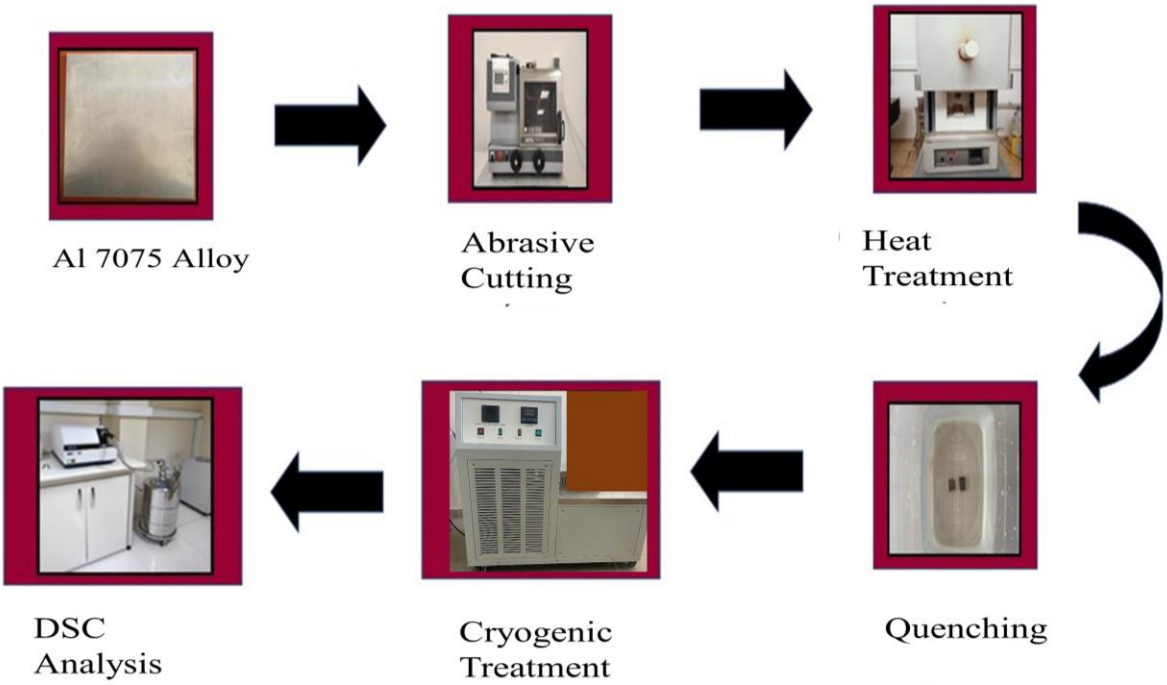

Figure 1. Flow chart of experimental studies (Deneysel çalışmaların akış şeması)

2. RESULTS (BULGULAR)

After the aging heat treatment, precipitates and phases begin to form in the 7075 aluminum alloy. The sequence of these phases as stated in the literature is shown in Figure 2. These phases form after certain temperatures. The dimensional difference, shape and distribution between GP zones largely depend on the alloy. If the size difference between Al and solute is small, spherical GP regions are formed, while large differences lead to the formation of plate-shaped GP regions. The sizes of GP regions are between 1-5 nm. GP regions are not a separate phase. They occur as a result of concentration differences. They are unstable structures that are completely compatible with the matrix [16,17]. These regions will then form metastable transition phases and finally the stable precipitate phase. The reason why transition phases meet before the stable equilibrium phase occurs; It is desired to reduce step by step the very large energy barrier that must be overcome for the formation of the equilibrium phase [18]. There are 2 types of GP zones. It is often seen when natural aging is done. It is consistent with the internal ordering of Zn and Al/Mg on the matrix lattice. GP II zones are formed by aging at temperatures above 70 °C after quenching at temperatures above 450 °C [19]. After the aging process from GPI to GPII and the growing GP regions, the ή phase begins to form in the microstructure. The main hardening phase phase ή (fine precipitates of metastable MgZn2 rich in Zn and Mg) is semi-compatible and meta-stable with the Al matrix, at least one surface of which is semi-compatible with the matrix [20].

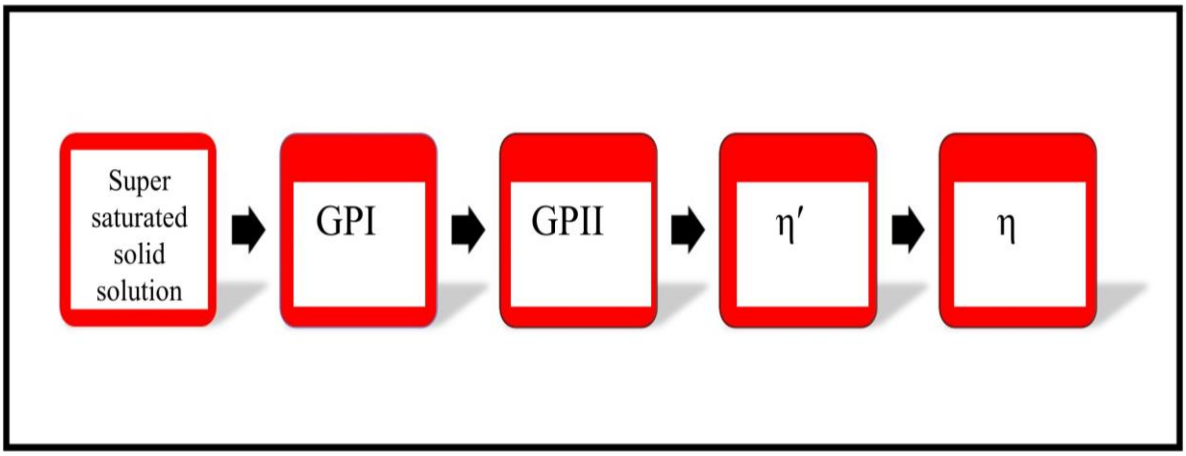

Figure 2. Sequence of phases formed by RRA heat treatment (RRA ısıl işlemiyle oluşan fazların sıralaması)

The transformation temperatures of GP, ή and η phases were determined by DSC analyses. The activation energies of the ή phase, which is the main strength-increasing phase, were calculated and the phase formation enthalpies were found using the Augis-Bennet and Kissinger equations. n the DSC graphs, it was observed that GP transformations occurred clearly in the samples subjected to cryogenic treatment. The significant transformations in the obtained GP regions showed that the cryogenic process created new nucleation points. The higher the number of GP sites, the more ή phases occur. Because as the diameter of the GP regions increases, there will be a transformation to ή phase. In other words, GP regions constitute the nucleation point for the ή phase [23,24].

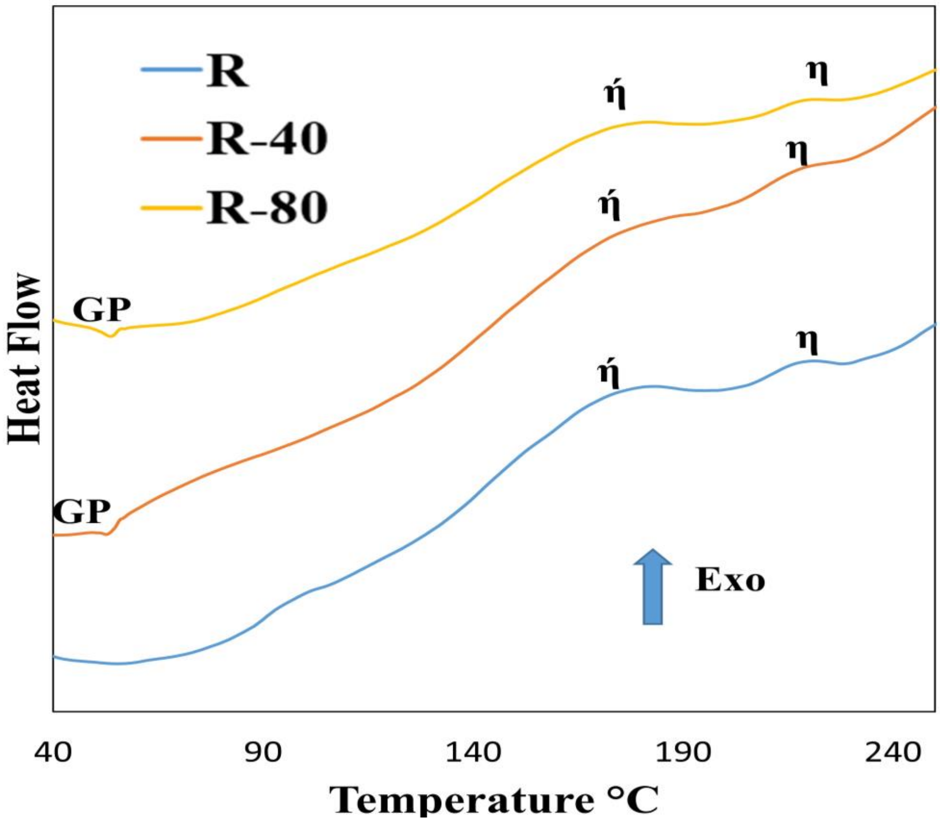

Figure 3. Transformations of phases formed by RRA heat treatment and cryogenic treatment (RRA ısıl işlemi ve kriyojenik işlem ile oluşan fazların dönüşümleri)

Kissinger equations [21];

In (B/Tx2) = −(Ea⁄RTx) + C

Multiplying the slope of the graph of ln(β/Tp2) versus 1000/Tp by the gas constant will equal the activation energy. Augis-Bennet equations [22]; In (B/Tx – T0) = −(Ea⁄RTx) + InK0

In this method, multiplying ln(β/(Tp-T0) by the slope of the line corresponding to (1000/Tp) and the gas constant gives the activation energy. Here;

Ea: activation energy

R is the gas constant (R=8.314 j/molK).

T is the temperature

B shows heating rates.

With these two equations, the ratio of the partial areas of the ἡ phase included in the DSC analyzes to the entire area, the slopes in the heating rates with the activation energies in Table 1 were calculated. It can be seen that the activation energy is highest for the ἡ phase of the R sample. The values in Table 1 were calculated by obtaining the graphs in Figures 3 and 4. It can perform the same calculations in both equations. The same calculations were made with different equations, the similarity of the results was tested and deviations were checked. For this reason, calculations were made with two different equations. In previous studies, the activation energies of the GP, ἡ and η phases formed during aging were calculated [25]. However, the kinetics of the phases formed after RRA have not been studied. The mechanism in the formation of phases with RRA and cryogenic process, the difference in growth and nucleation formation causes the change in activation energy.

Table 1. Activation energy of the ἡ phase formed in the samples (Numunelerde oluşan ἡ fazının aktivasyon enerjisi)

Samples	Slope (kissinger)	Slope (Augist bennet)	Average Activation energy (kj/mol)
R	-24,448	-24,242	201
R-40	-12,313	-13,237	102
R-80	-18,697	-18,492	155

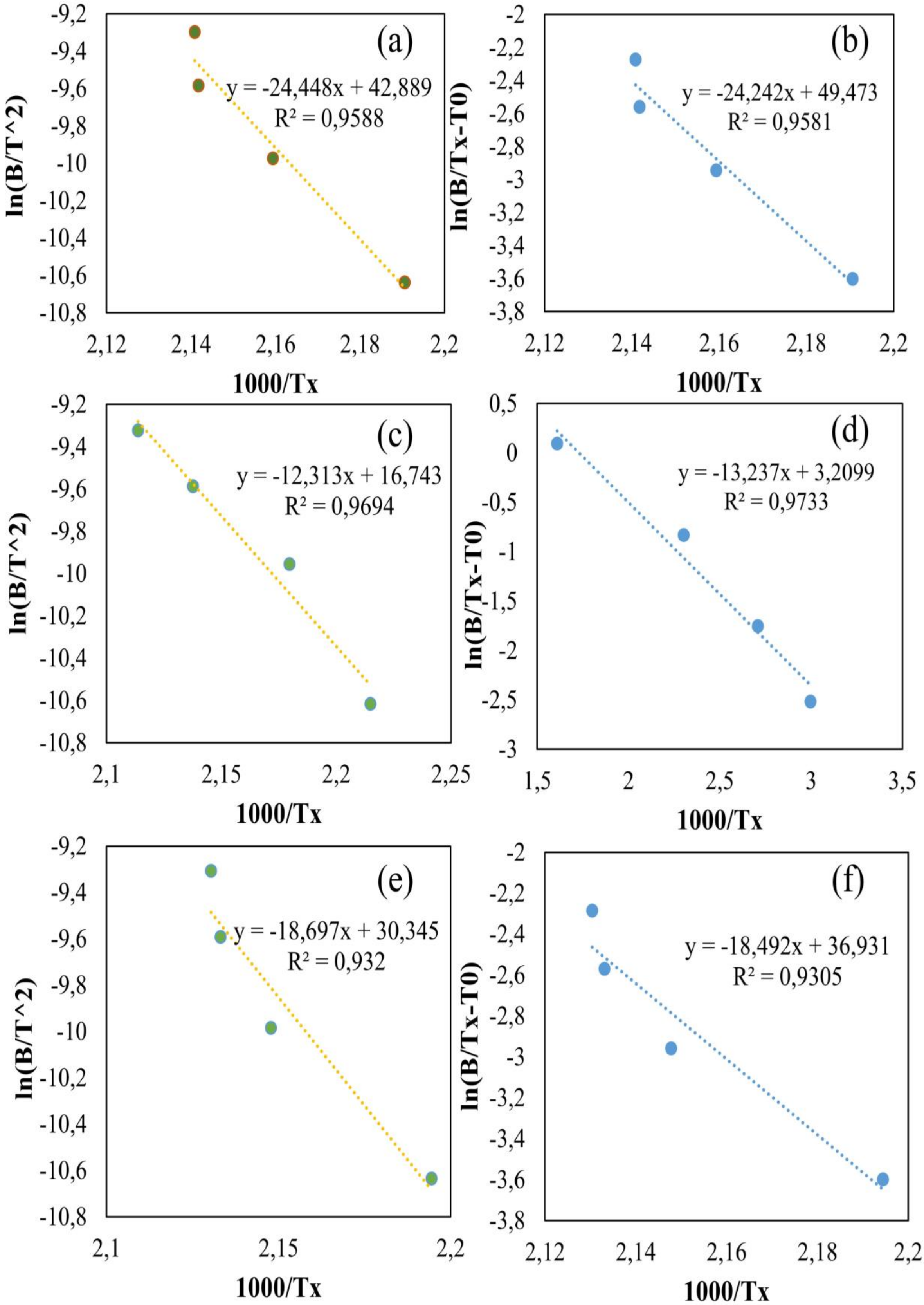

Figure 4. Slope graphs of the samples drawn with kissinger and Augis-Bennet equations (a) R sample kissinger (b) R sample Augis-Bennet (c) R-40 sample kissinger (d) R-40 sample Augis-Bennet (e) R-80 sample kissinger (f) R- 80 specimens of Augis-Bennet (Kissinger ve Augis-Bennet denklemleri ile çizilen örneklerin eğim grafikleri (a) R örneği Kissinger (b) R örneği Augis-Bennet (c) R-40 örneği Kissinger (d) R-40 örneği Augis-Bennet (e) R-80 örnek öpüşme (f) R- 80 Augis-Bennet örneği)

CONCLUSIONS (SONUÇLAR)

In this study, RRA and cryogenic process were applied to 7*** series aluminum alloy, and the transformations and activation energies of the phases formed for the first time in the literature were calculated with this heat treatment route. The results obtained showed that the cryogenic process created new nucleation points. In the DSC graphs, it was observed that GP transformations occurred clearly in the samples subjected to cryogenic treatment. The higher the number of GPs, the more ή phase will occur. Because when GP regions grow, they will turn into ή phase. Additionally, it has been observed that activation energy decreases when cryogenic treatment is applied. This decrease shows that the ή phase will form more easily through the diffusion mechanism.

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DECLARATION OF ETHICAL STANDARDS (ETİK STANDARTLARIN BEYANI)

The author of this article declares that the materials and methods they use in their work do not require ethical committee approval and/or legal-specific permission.

Bu makalenin yazarı çalışmalarında kullandıkları materyal ve yöntemlerin etik kurul izni ve/veya yasal-özel bir izin gerektirmediğini beyan ederler.

AUTHORS' CONTRIBUTIONS (YAZARLARIN KATKILARI)

***Gözde ALTUNTAŞ*:** She conducted the experiments, analyzed the results and performed the writing process.

Deneyleri yapmış, sonuçlarını analiz etmiş ve maklenin yazım işlemini gerçekleştirmiştir.

Bülent Bostan: *He* analyzed the results and performed the writing process.

Sonuçlarını analiz etmiş ve maklenin yazım işlemini gerçekleştirmiştir.

CONFLICT OF INTEREST (ÇIKAR ÇATIŞMASI)

There is no conflict of interest in this study.

Bu çalışmada herhangi bir çıkar çatışması yoktur.

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