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STATISTICAL ANALYSIS ON THE EFFECT OF THE SOLIDIFICATION RATE AND QUENCHING MEDIUMS ON MECHANICAL PROPERTIES IN ETIAL 221 ALLOY

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Abstract: In this study, the effects of different mold types and quenching mediums with different internal stresses on the mechanical properties of ETIAL 221 alloy supplied as a primer were investigated, and analyzed statistically. Firstly, the alloy was poured into the permanent mold (PM) then poured into sand mold with grain size 40/45 and 60/65 AFS. The specimens were subjected to three different T6 heat treatment quenching mediums that are water at room temperature (WRT), oil (OL) and boiling water (BW). The influences of differences in solidification rate due to cooling rates and differences in internal stress due to quenching mediums on mechanical properties were investigated. Additionally, the change in porosity was calculated according to Archimedes principle and obtained results were related to the mechanical properties. The results showed that permanent mold and boiling water quenching medium presented the best mechanical properties for the current study.

Keywords: ETIAL 221 alloy, Internal stress, Mechanical properties, Solidification rate, Quenching medium, Weibull analysis

ETİAL 221 Alaşımında Katılaşma Hızı ve Su Verme Ortamlarının Mekanik Özelliklere Etkisinin İstatistiksel Analizi

Oz: Bu çalışmada, primer olarak temin edilen ETİAL 221 alaşımında farklı kalıp türlerinin ve farklı iç gerilmelere sahip su verme ortamlarının mekanik özelliklere etkisi araştırılmış ve istatistiksel olarak incelenmiştir. Alaşımın ilk olarak kokil kalıba, daha sonra 40/45 ve 60/65 AFS boyutuna sahip kumlarla hazırlanmış kum kalıba dökümleri gerçekleştirilmiştir. Dökümden elde edilen parçalar oda sıcaklığında suda, yağda ve kaynayan suda su verme işlemleri ile üç farklı T6 çökelme sertleşmesine tabi tutulmuştur. Soğuma hızlarına bağlı katılaşma hızındaki farklılıkların ve su verme ortamlarına bağlı iç gerilme farklılıklarının mekanik özellikler üzerine etkisi araştırılmıştır. Ayrıca, porozitedeki değişim Arşimet prensibine göre hesaplanmış ve elde edilen sonuçlar mekanik özellikler ilişkilendirilmiştir. Sonuç olarak, kalıp türlerinden kokil kalıbın, su verme ortamlarından kaynayan suda su verme ortamının bu çalışma için en iyi mekanik özellik sergilediği görülmüştür.

Anahtar Kelimeler: ETİAL 221 alaşımı, su verme ortamı, katılaşma hızı, iç gerilim, mekanik özellikler, Weibull analizi

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1. INTRODUCTION

Aluminum and alloys, which are increasingly preferred day by day, are among the newest and most used metals in many industries. The reason of widespread use of aluminum alloys is that the material has low density, high corrosion resistance, and an improvable structure (Campbell, 2015). Properties of aluminum alloys can be enhanced by grain refinement (Campbell et al., Tiryakioğlu, 2006; Dispinar at al., 2009), modification (Uludağ et al., 2016; Uludağ at al., 2015; Uludağ et al., 2015; Uludağ et al., 2018; Uludağ et al., 2016) and degassing (Mostafaei et al., 2016) during casting and heat treatment (Casari et al., 2014) after casting. Aluminum-copper alloys are heat treatable materials. The presence of Cu up to 5% in these alloys causes solid precipitation in the structure. Mechanical properties such as hardness and toughness can be enhanced by natural or artificial ageing process in these materials (Kovarik et al., 2008).

T6 heat treatment is one of the heat treatment method applied to Al-Cu alloys (Speer et al., 2005). T6 heat treatment process consists of three stages: solutionizing, quenching, and ageing. The solution treated step is a process carried out at a point above solvus and below eutectic temperature and applied to dissolve the secondary phases at high temperatures. Quenching is applied by cooling to the room temperature directly from the solutionizing temperature in order to trap the dissolved secondary phases in the structure (i.e. supersaturated structure) and increase vacancy concentration. The alloy whose temperature is lowered to room temperature can be aged naturally by keeping it at room temperature for a long time or artificially by keeping it at temperatures higher than room temperature for a shorter duration. In the ageing process, the secondary phases precipitated in the grain boundary after the casting due to segregation, are nucleated at the vacancies in the structure. These secondary phases, which are homogeneously diffused in the vacancy, reduce the Gibbs energy of the system. Therefore, the final structure consists of finely distributed secondary phases within the grain. After this process, a considerable increase in strength is observed in the material (Costa et al., 2016; Emamy et al., 2015; Ibrahim et al., 2018; Magno et al., 2017).

The most preferred quenching medium in T6 heat treatment process is water at room temperature (WRT). One of the most critical steps of the T6 heat treatment is quenching mediums. Owing to the quenching process, high internal stresses occur in the alloy structure. As the cooling rate increases in the quenching phase, the internal stresses in the structure increase as well (Emamy et al., 2015). In addition, in quenching operations, with rapid cooling rates, distortion, cracking, and residual stress may occur in the material (Beitz, 1998; Mohamed and Samuel, 2012; Senatorova et al., 2016; Senatorova et al., 2002). Even if WRT is mostly used, it is preferred to use liquids with polymeric based additives, water at different temperatures, and oil to minimize internal stresses and distortion by reducing the Leidenforst effect (Croucher and Butler, 1981; Sverdlin et al., 1996; Totten and Mackenzie, 2000). Since distinct quenching mediums have different heat transfer coefficients, less internal stress may occur and the risk of distortion is can be minimized using these quenching mediums. In the literature (Guner et al., 2019; Tan et al., 2012), it is stated that keeping water temperature above 60-70 °C is effective on the mechanical properties after T6 heat treatment. This effect is more apparent in yield and tensile strength than in deformation (ductility). In a study where Al-7Si-0,4Mg alloy was used (Zhang and Zheng, 1996), after quenching in water at room temperature, it was stated that the structure consists of numerous needle-like α -Al matrix and coherent β ''-Mg₂Si particles. Particle size was measured at a diameter of about 3-4 µm and a length of 10-20 µm. It was observed that the quenching in water at 60 °C decreased particle density and also increased particle size. Besides, it was found that the fine silica particles, which are quite abundant in the structure, precipitated in the Al matrix. It was reported that low cooling rate reduced vacancy concentration in 7075 alloys by using three different quenching mediums (MacKenzies, 2002; Staley et al., 1972). Ammar et al. (Ammar et al., 2008) investigated the effects of alloying elements, solution time and quenching mediums on A413 alloy. They stated that hot water

quenching mediums could be preferable. It was reported that even though ductility of hot water quenched specimens decreased, alloy quality and strength of the material was enhanced.

On the other hand, one of the biggest problems which adversely affects the mechanical properties of aluminum alloys is the amount of porosity (Emadi et al., 1993; Uludağ and Dispinar, 2017; Uludağ et al., 2018). Although the reason of porosity formation is associated with hydrogen and solidification shrinkage (Laslaz and Laty, 1991; Lee and Gokhale, 2006; Sigworth and Wang, 1993), it is claimed that the main reason is the oxide layer formed on the surface of the liquid alloy which is broken down and submerged in the liquid during mold filling and thus forms porosity during solidification (Dispinar et al., 2010; Dispinar and Campbell, 2004; Dispinar and Campbell, 2006; Dispinar and Campbell, 2011). It is reported that dissolved gas content and solidification shrinkage are not the main factors but they only trigger pore formation. In an alloy produced by casting, the casting defects influence the mechanical properties far more than the heat treatment process parameters. While the solidification rate shapes the microstructure, it also determines porosity morphology (Caceres et al., 2002; Uludağ et al., 2018; Yamamura et al., 2001). As the solidification rate decreases, the size of porosity increases while number porosity increases as well. The larger the size of porosity in the structure, the more likely it is that the porosity will cause cracking and reduce the mechanical properties (Dispinar and Campbell, 2011).

In this study, T6 heat treatment of ETIAL 221 in three different quenching mediums was investigated. The influence of mold materials on heat treatment was evaluated by using a permanent mould and two sand moulds with different sand sizes: 40/45 and 60/65 AFS. The results were analyzed statistically.

2. EXPERIMENTAL PROCEDURE

Lorem An Al-Cu alloy that is included 4.5% Cu content and called as ETIAL 221 was selected for the study. The alloy was provided from ETİ Alüminyum Company in Turkey. The chemical composition of the alloy is given in Table 1.

Si	Fe	Cu	Mn	Mg	Zn	Ti	Ni	Pb	Al
0.30	0.30	4.50	0.10	0.05	0.10	0.15	0.10	0.05	Rem.

Table 1. Chemical composition of the alloy that was used in the study

An experimental study was carried out in three stages which are casting, T6 heat treatment and analysis of the results. An electrical furnace and SiC crucible were used in the casting process. The melt was poured into the tensile test molds which have 13 mm diameter and 150 mm length at 775 °C of pouring temperature. Three different mold types were used in the study. The mold types are permanent mold (PM), sand mold prepared by 40/45 AFS and 60/65 AFS sand. Dimensions of the tensile test mold are given in Fig. 1.



Dimensions of tensile test mold

At T6 heat treatment stage, three different quenching mediums were selected: Water at Room Temperature (WRT), Oil (OL) and Boiling Water (BW). Solution temperature was selected as 537 °C, artificial aging temperature was selected as 195 °C. Optimum duration of solution was preferred as 30 minutes for water at room temperature medium and 60 minutes for oil and boiling water mediums. Additionally, the optimum duration of artificial aging was chosen as 12 hours for samples that were solved in water at room temperature and 16 hours for samples that were solved in water (Zhang and Zheng, 1996). The schematic presentation of the T6 heat treatment which was used in the study is given in Fig. 2. Also, all the parameters of the study are summarized in Table 2.



Figure 2:

Schematic presentation of the optimum parameters for the T6 heat treatment: a) for water at room temperature (WRT) medium and b) for oil (OL) and boiling water (BW) mediums

	Quenching Mediums							
	Without any Heat Treatment (WHT)	Water at Room Temperature (WRT)	Oil (OL)	Boiling Water (BW)				
Permanent mold (PM)		30 minutes of solution	60 minutes of solution	60 minutes of solution				
40/45 AFS sand mold (40/45 AFS)	40/45 AFS sand mold Yes 40/45 AFS)		and 16 hours of artificial	and 16 hours of artificial				
60/65 AFS sand mold (60/65 AFS)	•	aging	aging	aging				

Table 2. Experimental conditions

Firstly, hardness and tensile test results of the samples were measured after T6 heat treatment. Secondly, hardness, ultimate tensile stress (UTS), yield stress (YS), Elongation (e %) and Toughness were examined. Lastly, all results of the experimental study were analyzed statically by using a statistical analysis software (Minitab).

3. RESULTS AND DISCUSSON

The effect of different mold types on solidification rate and the effect of different quenching mediums on internal stress of the alloy were investigated, the findings were categorized under four subheadings. Firstly, the effects of solidification rate and quenching mediums on hardness were discussed. Then, tensile test results depending on solidification rate and quenching mediums were presented. Thirdly, the volumetric porosity was evaluated which was measured according to the Archimedes principle and the impacts of volumetric porosity on mechanical properties were compared. Finally, the mechanical properties depending on solidification rate and quenching mediums were analyzed statistically and discussed.

3.1. Hardness Sensitivity

The casted parts produced using 60/65 AFS, 40/45 AFS, and permanent mold were quenched in three different mediums and hardness of the parts was presented in Fig. 3. When results analyzed, it was observed that there is a significant change in hardness of untreated specimens depending on molding types (Fig. 3a). While the highest hardness value was measured from parts casted by permanent mold, the lowest hardness was measured from parts cast in 60/65 AFS sand mold. Also, the margin of error between measured hardness values is the lowest in the permanent mold. Although these results in PM can be attributed to the fact that the grain size is finer and therefore it prevents dislocation movements (Pešička et al., 2003), it is suggested that the size and distribution of the porosity in fine-grained structure was also a major factor (Zhilyaev et al., 2003). The porosity formed in small-sized morphology in fine-grained structure adversely affects the hardness values. When the hardness change depending on quenching mediums is examined, it is seen that the lowest hardness values were obtained in WRT. (Fig. 3b). Hardness variations depending on mold types show similar trend for three mediums. There are no significant effects of quenching mediums on cast parts in 60/65 AFS and 40/45 AFS sand molds. The hardness values for such two molds are similar in three quenching mediums. The hardness of cast parts in PM were the highest in all three mediums. When the results evaluated in terms of the changes in standard deviation are examined, it is seen that there

are significant differences depending on quenching mediums. Whilst the results with the lowest standard deviation belongs to BW (Fig. 3d), the highest standard deviation is found in the oil quenching medium (Fig. 3c). Considering both hardness and deviations in hardness, it can be said that the optimum hardness value is obtained in PM casting and BW T6 heat treatment combination.



Changing of hardness values in quenching mediums depending on mold types: a) Without any heat treatment (WHT), b) Water at room temperature (WRT), c) Oil (OL) and d) Boiling water (BW)

3.2. Tensile Test Results

The results of the tensile tests depending on molding type and quenching mediums are shown in Fig. 4. It can be concluded from all the graphs that the mechanical properties vary depending on molding types. While PM presented the best results, 60/65 AFS sand mold shows the worst results, a more detailed examination of the findings may be useful for better understanding. When the tensile test results of untreated specimen are investigated, it is seen that there are no significant changes in ultimate tensile strength (UTS) and yield strength (YS) values, while the considerable change was obtained in elongation (e %) and toughness values depending on molding types. The greatest change was observed in PM casting. There was a little increase in parts casted in 40/45 AFS as compared with the parts casted in 60/65 AFS.



Tensile test results depending on mold types: a) UTS, b) YS, c) e % and d) Toughness

When specimens quenched in WRT are examined, it is seen that highest value for the ultimate tensile strength belongs to PM and for yield strength, it belongs to 40/45 AFS sand mold. For elongation and toughness, it belongs to PM. Although it may be said that the highest value for yield strength was obtained in 40/45 AFS sand mold, there was no significant difference between in yield strength for three mold types. In this manner, it can be said that the best mold type for WRT is permanent mold. Given the results of quenching in the oil, it is understood that the variation of all heat treatment test results is similar to that of water quenching medium. To make a general comparison, it can be said that the permanent mold shows the best results for this medium. When the results of quenching in BW are analyzed, it is seen that there is an increase in each property of PM. Lastly, when the tensile test results depending on quenching mediums for PM is considered (whilst the highest value in the ultimate tensile strength belongs to the BW quenched specimens), the lowest value belongs to WRT quenched specimens. The change in the yield strength values is similar to the change in the ultimate tensile strength values. However, the effect of the quenching medium on the elongation values has a different story. The highest value belongs to the specimens quenched in WRT and the lowest value belongs to the samples quenched in OL. In the elongation results, an increase was observed for WRT quenching medium compared to the untreated specimens while a decrease occurred for BW and OL mediums. The change in water temperature causes an

increase in the ultimate tensile strength and decrease in the elongation which is in good agreement with the results of Ammar et al. (Ammar et al., 2008). An increase was observed in the toughness values for three quenching mediums. The highest increase was seen in WRT and BW mediums and the rate of increase is almost equal to each other. In oil quenching medium, there was a slight increase compared with other mediums. When a general evaluation is considered, it is concluded that the toughness is a function of the temperature where WRT quenching medium is more suitable than oil quenching medium.

3.3. Volumetric Porosity

The density of untreated and T6 heat treated specimens in WRT were calculated according to Archimedes principle and the volumetric porosities were determined. The results are given in Fig. 5. After heat treatment, there was a slight increase in the density of specimens. This increase caused a decrease in porosity. When it is taken into account that ageing is effective on vacancy concentration in the structure (Sjölander and Seifeddine, 2010), the decrease in the porosity depending on heat treatment is an expected result. When porosity ratios are compared according to the mold types, it can be said that the lowest porosity is obtained for permanent mold.



Changing in volumetric porosity in without of any heat treatment (WHT) and water at room temperature (WRT) medium depending on mold types

The relation between volumetric porosity, hardness, ultimate tensile strength and toughness for WHT and WRT specimens was investigated and results were given in Fig. 6. It is seen that as the volumetric porosity increase, the mechanical properties decrease.

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Figure 6:

Relationship between volumetric porosity and mechanical properties for a) Without any heat treatment (WHT) and b) Water at room temperature (WRT)

3.4. Statistical analysis

All mechanical properties obtained from experimental studies were analyzed statistically and shape factor (Weibull modulus) and scale parameter (characteristic alpha) were calculated by using a statistical analysis software. Weibull analysis of the UTS is given in Fig. 7. As seen in the figure, the mold types and quenching medium have an important role on the UTS values. Differences between the average values of UTS is similar to the discussion above in the first subheading of this article. The statistical analysis show that the most reliable and stable value belongs to WHT samples produced with 40/45 AFS sand mold as 39. Next three reliable values are 28, 19 and 18 for BW samples produced with 40/45 AFS sand mold, WHT samples produced with PM and BW samples produced PM, respectively. When the scale parameter are compared, it can be reported that the characteristic alpha values are 132, 154, 201 and 259 MPa for WHT-40/45 AFS, WHT-PM and BW-PM respectively. 259 MPa is a fairly great value in comparison with 154, 132 and 201 MPa. Therefore, it can be concluded that the optimum parameter for UTS is boiling water of quenching medium and permanent mold in terms of reliability and reproducibility. This parameter can be followed by the results of boiling water and 40/45 AFS sand. On the other hand, the first and second worst values were obtained from WHT samples produced with 60/65 AFS sand and WRT samples produced with 60/65 AFS sand.



Weibull analysis of the UTS results obtained from all experimental parameters

Results of the Weibull analysis of elongation at fracture values obtained from tensile tests are given in Fig. 8. It can be said that the samples poured in 40/45 AFS sand mold and heat treated using WHT has the most reliable values. Conversely, the samples poured in PM and heat-treated BW has the worst values. The data obtained from Weibull analysis were evaluated in detail and the results are presented as an optimum parameter in terms of elongation. The optimum parameter for current study is found to be PM-WHT for the elongation due to high scale parameter and average shape parameter.



Weibull analysis of the elongation (%) results obtained from all experimental parameters

In addition, the results of YS were analyzed by using statistical analysis which is given in Fig. 9. Similar to UTS, the highest average YS value is obtained in the samples that were produced in PM mold and heat treated in BW medium. However, when the shape parameters that represent reliability and stability of results are evaluated for all conditions, it can be said that although PM-BW parameter has the highest average YS value, it is the worst parameter in terms of reliability with 7.07 of shape factor. The highest three experimental parameters for reliability are 40/45 AFS-WHT, 60/65 AFS-OL and 60/65 AFS-WHT with 59.21, 51.26 and 35.86 respectively. And, the highest three experimental parameters for scale that means maximum average value are PM-BW, 40/45 AFS-OL and 40/45 AFS-BW with 201.2, 175.6 and 174.1 respectively. If the optimum parameters for a suitable application is considered, it can be concluded that 60/65 AFS-OL parameter can be preferred.



Weibull analysis of the YS results obtained from all experimental parameters

Toughness has a vital factor to describe mechanical properties of an alloy, particularly for material selection applications. Therefore, the toughness is mostly preferred to measure by using UTS, YS and *e* %. It can be used to characterize an alloy to be used in any application (Sokolowski et al., 1995). Statistical analysis of the toughness results obtained from experimental parameters of this study were carried out. The results are given in Fig. 10. When looking at the figure, the first conspicuous difference of the data of scale parameters is that of PM casting in all quenching mediums have the highest toughness value. It can be concluded easily from this difference that the cooling rate of solidification has an important effect on the toughness. The second difference is in shape. The highest shape values of the toughness in all quenching mediums are found in samples poured in 40/45 AFS sand mold. Even though samples of 40/45 AFS sand mold has good reliability and stability, they display toughness value much less than PM's. It can be suitable to find optimum condition that the shape parameters of quenching mediums of samples of PM are sorted; i- WHT (6.43), ii- WRT (3.72), iii- OL (2.57) and iv- BW (2,5). It can be a critical conclusion that PM-WRT is the optimum condition to reach the acceptable toughness value as high as possible and good reliability.



Weibull analysis of the Toughness results obtained from all experimental parameters

It was stated that the mold types were used for different cooling rates and three different quenching mediums were selected to evaluate the internal stress in samples. The cooling rate can be labeled as 1, 2 and 3 for 60/65 AFS, 40/45 AFS and PM respectively. Additionally, internal stress of quenching can be labeled as 1, 2 and 5 for BW, OL and WRT respectively. It was presented that calculations of internal stress of oil and boiling water mediums are close to each other but internal stress of the oil medium is little bit higher than the internal stress of boiling water (Speer et al., 2005; Zhang and Zheng, 1996) than oil and boiling water. Statistical analysis was carried out by using counter plot ingredient of the software (Minitab) which is given in Fig. 11.



Figure 11:

Change in the hardness with internal stress of quenching and cooling rate

As seen in Fig. 11, the hardness of the alloy that is heat treated is affected by quenching mediums because of internal stress and mold types due to solidification rate. As the internal stress increases, the hardness of the alloy tends to decrease. On the other hand, the hardness of the alloy improves with increased solidification rate which can be provided by rapid cooling. It can be said that the minimum internal stress and maximum solidification rate should be applied for high hardness value. PM-BW condition of the current study appears to be suitable for these requirements. Besides hardness values, mechanical properties of the alloy after heat treatment was also analyzed and change in the mechanical properties depending on the cooling rate and internal stress of quenching together is given in Fig. 12 for UTS and e % and Fig. 13 for YS and toughness.



Figure 12:

Change in a) UTS and b) elongation (%) with internal stress of quenching and cooling rate

In general terms, it is known that while UTS is increased, elongation is mostly decreased in most engineering materials (Hwang et al., 2008; Rana et al., 2012). Trend of the UTS and elongation results of this study is similar to conclusions of the researches (Hwang et al., 2008; Rana et al., 2012). While UTS value of a sample is increased, elongation of the sample tends to decrease. If the results of these two properties of the alloy are evaluated in terms of both solidification rate and internal stress of quenching, Fig. 12 can be examined in detail. As the internal stress of quenching is increased, UTS of the alloy decreases while elongation increases. On the other hand, both UTS and elongation of the alloy improve with the increasing

in solidification rate. It can be concluded from these results that the optimum conditions for UTS and elongation is found to be as PM-BW and PM-WRT respectively.



Figure 13:

Change in a) YS and b) Toughness with internal stress of quenching and cooling rate

When the results of the analysis for YS of the alloy are inspected (Fig. 13a), it can be seen clearly that while YS increases as the solidification rate increases, it decreases as internal stress of quenching increases. For the toughness of the alloy (Fig. 13b), it can be said that it is increased with increased solidification rate. As a general result for the toughness of the alloy, it can be concluded that the toughness of the alloy is achieved to be the highest in two conditions which are minimum internal stress and maximum solidification rate (PM-BW) and maximum internal stress with maximum solidification rate (PM-WRT).

4. CONCLUSION

The current study aims to investigate how the properties of ETIAL 221 alloy changes under different mold types and different quenching mediums. The conclusions of the study based on the effect of solidification rate and internal stress due to quenching medium on mechanical properties can be summarized as follow:

- Volumetric porosity decreases as solidification rate increase (from 60/65 AFS sand mold to permanent mold) and this results in improved mechanical properties.
- Hardness of the alloy can be increased with permanent mold casting followed by boiling water for quenching which results in low internal stress. On the other hand, the hardness of the alloy improves with increased solidification rate which can be provided by rapid cooling. It can be said that the minimum internal stress and maximum solidification rate should be applied for the achievement of high hardness value. PM-BW condition of the current study provides the suitable condition for these requirements.
- The highest values of UTS have been obtained from boiling water parameters as quenching medium. Oppositely, the worst values of UTS have been obtained from water at room temperature as quenching medium. On the other hand, it can be concluded that the optimum parameter for UTS is boiling water of quenching medium and permanent mold in terms of reliability and stability.
- While water at room temperature medium has a positive effect on the elongation, oil medium has a negative effect. The optimum parameter for the current study is found to be PM-WHT for elongation due to high scale parameter and average shape parameter.

- Change in the yield stress of the alloy is similar to UTS's results. For the optimum parameters for a suitable application, it can be advised that 60/65 AFS-OL parameters can be preferred when a reliable and high yield stress value is required. While the optimum UTS value was obtained as 200 MPa, the optimum YS value was obtained as 140 MPa in this work.
- The toughness of the alloy can be the highest for two conditions which are minimum internal stress and maximum solidification rate (PM-BW) and maximum internal stress with maximum solidification rate (PM-WRT).

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