

# Thermal Shock Effect on Strength Loss Properties of Rock Materials: An Experimental Study on Thermal Fatigue Durability

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## Abstract

Four different types of rock materials from Eastern Black Sea Region of Turkey were investigated to determine their change in porosity as well as the degree of cracking, fracturing, disintegration and strength loss under heating-cooling cycles with variations in temperatures up to 300 °C. Cooling time and thermal strains of the rock specimens were also determined in this study. Totally, sixty rock core samples were tested to evaluate their physico-mechanical and mechanical properties. Thermal cycling was found to lead for increase in the porosity of the rocks, making new cracks, particle disintegration for two types of tested rocks and considerable losses in uniaxial compressive strength values of all the rock materials tested in this study. The purpose of this study is to investigate immediate changes in temperature values rather than step by step heating up to a target temperature. For instance, rock samples were directly put in 250 °C stove from a -50 °C cabin in the last and fifth thermal cycling. After the heating process, specimens were cooled in water and air. This study aims to be a usable reference to establish a thermal change procedure and improve a new testing method for determination of thermal fatigue durability of rock materials.

## Keywords

Strength Loss, Rock Strength, Thermal Treatment, Thermal Fatigue

## Termal Şok Etkisinin Kaya Malzemelerinin Dayanım Kaybı Özellikleri üzerindeki Etkileri: Termal Yorulma üzerine Deneysel bir Çalışma

## Özet

Doğu Karadeniz bölgesinden dört farklı kaya malzemesinin, 300 °C'ye kadar değişen sıcaklık değişimlerine sahip termal döngüler altında porozite değerlerindeki değişim, çatlama, parçalanma ve dayanım kaybı özellikleri incelenmiştir. Numunelerin soğuma süreleri ve soğuma esnasındaki birim deformasyonları da bu çalışma kapsamında belirlenmiştir. Toplamda altmış adet karot numunesi test edilmiştir. Elde edilen sonuçlara göre, döngü sayısı arttıkça porozite değerlerinde artış, dayanım değerlerinde düşüş yaşanmakta olduğu, kaya malzemesi türüne bağlı olarak çatlamlar ve parça kopmaları şeklinde bozunmaların olabildiği görülmüştür. Çalışmadaki termal döngülerde ani sıcaklık değişimleri incelenmiştir. Örneğin, beşinci ve son döngüde numuneler 250 °C etüvden alınarak doğrudan -50 °C sıcaklıktaki soğutma kabinine alınmıştır. Isıtma işleminden sonra numuneler suda ve havada farklı ortamlarda soğutulmuşlardır. Bu çalışma, kaya malzemelerinin termal yorulma karşısındaki dirençlerinin değerlendirilmesi üzerine ısıtma-soğutma işlemleri prosedürün belirlenmesi ve geliştirilecek yeni bir metoda yönelik çalışmalara kullanışlı bir referans oluşturmayı amaçlamıştır.

## Anahtar Sözcükler

Dayanım Kaybı, Kaya Dayanımı, Termal Döngü, Termal Yorulma

## 1. Introduction

The temperature is a well-known factor that significantly change the strength and deformability properties of rock materials. Both compressive and tensile strength values decrease with an increase in temperature. Rock materials are considerably expanded and softened in case of being highly heated, which is a reason for the strength loss and decrease of the modulus of elasticity values, as well (Mahmutoglu 1998; Mahmutoglu 2006; Akbay et al. 2014; Török and Török 2015).

Depending on the heating temperature, cooled rock materials can conserve their strength values. In other case, rock materials are plastically deformed having micro-cracks after cooling, as a result of high thermal strain levels. Number of thermal cycles is an important parameter for having plastic deformation after cooling and strength loss due to the thermal stresses (Papay and Török 2018). For many of rock materials, thermal strain levels can be much higher than the mechanical strain levels had before heating and a notable softening that makes a big change in deformability properties are seen under various temperatures such as degrees over 300 °C.

In this study, different rock materials were heated to increase temperature values for up to 300 C° to determine strength loss values and changes in physical properties under thermal cycles. As a result of thermal stresses, micro or visible macro cracks that make notable strength losses occur depending on various factors such as temperature, duration of heating, cooling details and some rock properties like mineralogical content, grain size, porosity, micro cracks before the heating process. As a reason for having strength loss under being heated and cooling, variations in strains of different minerals make cracking at the mineral contacts and propagation of existing cracks (Wang and Hao 2017; Sygala et al. 2013). That situation makes physical weathering of rock materials. Additionally, chemical weathering under high temperatures should be noted as another considerable reason for having strength loss due to the thermal changes. As same in the physical weathering, mineralogical content of the rock materials and temperature level are determinative parameters for chemical weathering. Because the temperatures in this study are generally not critical for the start of chemical weathering, the physical weathering was focussed on in this study, rather than chemical weathering.

In case of an increase in the cooling rate, the possibility of observing major cracks increases in comparison with the case of slow cooling. Temperature and heat capacity values of the cooling environment change the cooling rate and cooling effect on cracking (Isaka et al. 2018; Browning et al. 2016). In this study, it is aimed to investigate the effect of different thermal cycles including different cooling conditions in air and water on change of physico-mechanical and strength loss properties of various rock materials.

## 2. Materials and Methods

Totally, sixty rock core specimens were tested in this study. Limestone, Andesite, Dacite, Basalt and a Chalcopyritic copper ore type different rock materials from North-Eastern Region of Turkey were used in tests for determining thermal strains under different temperatures, reversal thermal deformation capacity, cooling time, changes in porosity values and the strength loss. According to the International Society for Rock Mechanics and Rock Engineering (ISRM) suggestions, core cutters with the diameter of 54.7 mm (NX size) were used and all of the rock core specimens were cut to have a same length to diameter ratio of 3 by using sawing machines (Figure 1). The end faces of the rock cores were smoothened and made perpendicular to the sample axis within 0.05 mm using comparator. Loading rate was selected to be 0.5 MPa/sec during the uniaxial compressive strength (UCS) test, according to the ISRM suggestions (ISRM 2007). A hydraulic press with the maximum load capacity of 300 kN was used in the UCS test (Figure 2).

For determination of the apparent porosity, core samples were immersed in water for a day to make saturation. As the specimens were removed from water bucket, their surfaces are dried by gently wiping with tissue. Specimens were weighed using a digital scale with a sensitivity of 0.01 g. After determination of saturated rock weights, the core specimens were put in 105 C° stove to dry specimens for 24 hours, then the dry masses were determined to evaluate the evaporated water weight and volume of voids. To accurately and sensitively calculate volumes of specimens, lengths and diameters of each of rock core specimens were separately measured using a digital vernier calliper. Following the method suggested by ISRM (2007), porosities of rock core specimens were carefully determined before and after heating cycles. Testing environment and apparatus used for porosity tests are shown in Figure 3.

Temperature changing procedure for heating cycles is given in Table 1. In this study, two different cooling processes were carried out under cooling in air and cooling in water conditions, to compare different cooling environments. Temperature changing procedure was same for specimens cooled in both air and water, that were heated together in the stove. Relevant methodological details are given in the following subtitles.

Table 1: Thermal change procedure

Cycle name	Cooling Temperature Before Heating (C°)	Heating Temperature (C°)	Cooling Temperature After Heating (C°)
Cycle 1	20	120	20
Cycle 2	20	170	20
Cycle 3	20	220	20
Cycle 4	0	250	20
Cycle 5	-50	250	20



Figure 1: a) Rock coring, b) core cutting by using sawing machine



Figure 2: Uniaxial compressive strength test

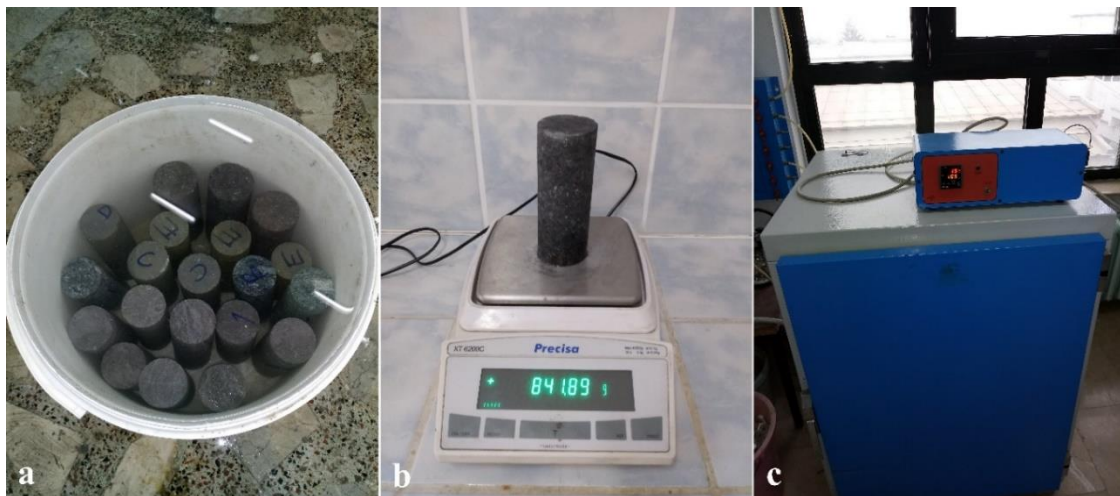


Figure 3: Porosity test: a) saturation of specimens in water, b) weighing, c) stove



## 2.1. Cooling in air condition

For heating and cooling processes, a stove with maximum temperature capacity of 260 °C and a freezing cabin with a lowest temperature capacity of -60 °C were used in this study. Totally, five cycles were applied including 17 hours heating in stove and cooling stages in each cycle. Specimens were cooled in air at room temperature in laboratory (~20 °C). In addition, specimens were respectively cooled in 0 °C and - 50 °C cabin before heating processes in 250 °C stove in fourth and fifth cycles. The cabin cooling stage duration in fourth and fifth cycles was 2 hours after cooling in room temperature. In each heating cycle, temperature change was increased for 50 °C in comparison with that of a previous one. In the last step, specimens were taken from -50 °C cabin and exposed the temperature increase of 300 °C. As seen in Figure 4, stove and freezing cabin were near to each other and there was no time loss between cooling and heating processes.

After each of heating processes, strains of the specimens during cooling in air at room temperature were measured using a dial gauge as seen in Figure 5. It should be noted herein that strains of specimens could be rapidly measured at the end of the heating process since the dial gauge setup and stove were in the same room. It took only several seconds to start strain measurement after taking specimen from stove. Shortening deformation under cooling was read and noted for changes in time. It should be noted herein that same specimens were tested in the strain measurements after heating processes to be able to accurately compare changes due to the thermal cycles. After completion of the strain measurement, specimens were kept at room temperature until the start of next thermal process. Totally, 20 core specimens were applied thermal cycles under the condition of cooling in air. After the last thermal cycle, uniaxial compressive strength (UCS) test was carried out to evaluate the heating-cooling effect on the strength loss property. Additionally, USC test on other 20 core specimens were performed without applying thermal processes to determine the strength values of rock materials before heating and strength losses due to the thermal cycles. The rock samples used in this study were selected carefully after visual observations to check core samples for having no anisotropy, fractures, cracks, fill joints etc.



Figure 4: Freezing (cooling) cabin and stove in the laboratory

## 2.2. Colling in water condition

The tap water at the room temperature was used for cooling. The heating procedure of the specimens cooled in water was same with that of the specimens cooled in air. The procedure in Table 1 was applied for both of the cooling conditions. The cooling strains of the specimens in water could not be measured. Specimens were put in water buckets for cooling process for half an hour. Later on, specimens were waited for nearly six and half hours in air for the next thermal process, before second and third thermal cycles. As same with the cooling in air condition, specimens were respectively cooled in 0 °C and - 50 °C cabin before heating processes at 250 °C in fourth and fifth cycles. As the cabin cooling process duration was 2 hours, specimens were waited under air at room temperature for four and half hours before cabin cooling process in fourth and fifth cycles. Heating in stove duration was 17 hours in each thermal cycle.



Figure 5: Cooling strain measurement using dial gauge

### 3. Results

The results obtained from porosity tests for heated and unheated specimens are given in Table 2. Table 3 includes cooling time to stop shortening versus thermal strain data obtained from dial gauge measurements. Additionally, thermal strain levels under different thermal cycles are shown in Figure 6. Because of having no ability to measure heated specimen lengths, strain levels given in Table 3 and Figure 6 are the ratio of deformations during the cooling to lengths of the specimens in the room temperature. Hence, it can be assessed as a reverse measurement of approximate thermal expansion strain with an initial length under the room temperature. The strength loss data is given in Table 4 including UCS test results for specimens before and after thermal cycles. Andesite specimens were seen to have visible cracks and one of them cooled in air had a visible disintegration. Moreover, two andesite specimens cooled in water could not be used in the UCS test because of major disintegration (Figure 7). Andesite specimens with disintegration problem were not preferred to be used in the UCS test because of the cross-sectional change. Limestone specimens cooled in water had also visible and minor cracks at the end of thermal cycles. As parallel to the observations, the biggest strength loss was found to be in the andesite specimens that were followed by the limestone specimens. Except of the hard chalcopyrite ore from Akarsen mine, rock specimens had not ignorable strength loss and increase in porosity values. Effect of thermal cycles on strength losses is graphically given in Figure 8.

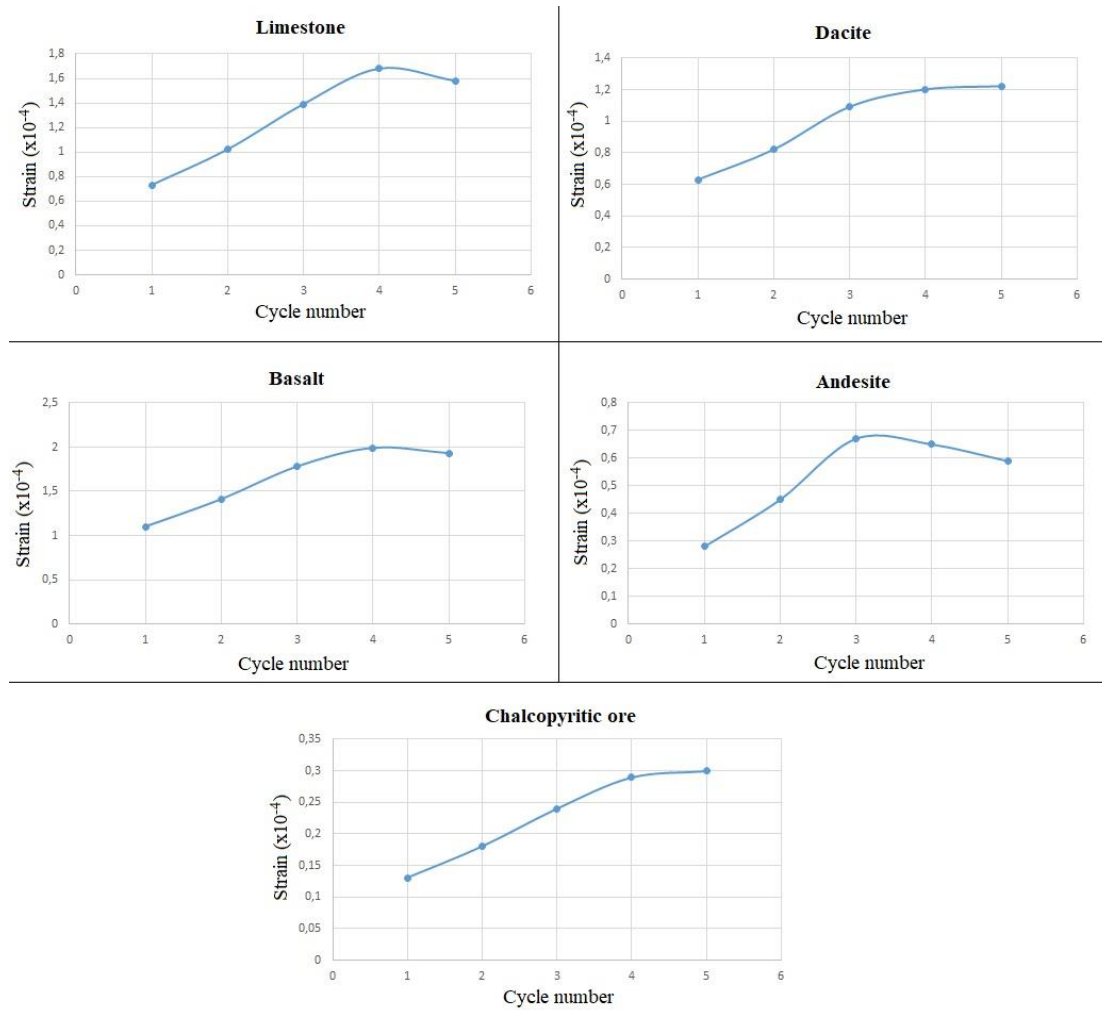


Figure 6: Cooling strains under different thermal cycles (cooling in air condition)

Table 2: Porosity test results

Rock type	Before Thermal Cycles			After Thermal Cycles (AC)			After Thermal Cycles (WC)		
	Porosity	S.D.	N.	Porosity	S.D.	N.	Porosity	S.D.	N.
Limestone	0.032	0.007	4	0.045	0.006	4	0.059	0.009	4
Dacite	0.028	0.005	4	0.033	0.004	4	0.041	0.005	4
Basalt	0.034	0.009	4	0.039	0.007	4	0.046	0.006	4
Andesite	0.063	0.011	4	0.094	0.012	3*	0.120	0.014	2**
Calc. Ore	0.039	0.004	4	0.046	0.005	4	0.058	0.007	4

S.D.: Standard deviation, N: Specimen number, \* one disintegrated specimen, \*\* two disintegrated specimens not tested

Table 3: Strain ( $\varepsilon$ ) measurements

Rock type	Cycle 1		Cycle 2		Cycle 3		Cycle 4		Cycle 5	
	$\varepsilon$ ( $\times 10^{-4}$ )	t (min)	$\varepsilon$ ( $\times 10^{-4}$ )	t (min)	$\varepsilon$ ( $\times 10^{-4}$ )	t (min)	$\varepsilon$ ( $\times 10^{-4}$ )	t (min)	$\varepsilon$ ( $\times 10^{-4}$ )	t (min)
Limestone	0.73	21	1.02	29	1.39	35	1.68	42	1.58	39
Dacite	0.62	20	0.81	32	1.09	45	1.20	54	1.22	55
Basalt	1.10	16	1.41	22	1.78	27	1.99	33	1.93	32
Andesite	0.28	15	0.45	20	0.67	24	0.65	28	0.59	24
Calc. Ore	0.13	30	0.18	39	0.24	50	0.29	56	0.30	56

t: approximate time to stop cooling strain

Table 4: Uniaxial compressive strength (UCS) test results

Rock type	Before Thermal Cycles			After Thermal Cycles (AC)			After Thermal Cycles (WC)		
	UCS (MPa)	S.D. (MPa)	N.	UCS (MPa)	S.D. (MPa)	N.	UCS (MPa)	S.D. (MPa)	N.
Limestone	32.7	1.4	4	28.2	1.9	4	25.4	1.5	4
Dacite	64.0	3.8	4	62.1	3.5	4	60.3	2.7	4
Basalt	79.8	3.7	4	75.0	4.0	4	70.5	3.9	4
Andesite	45.5	3.2	4	35.9	3.8	3*	25.6	4.0	2**
Calc. Ore	82.4	2.9	4	80.7	3.3	4	78.2	3.5	4

S.D.: Standard deviation, N.: Specimen number, \* one disintegrated specimen, \*\* two disintegrated specimens not tested, AC: Cooling in air, WC: Cooling in water



Figure 7: Some crack shapes observed from this study: a) Narrow cracks, b) a major cracking, c) disintegration, d) disintegration by a long crack all through the specimen

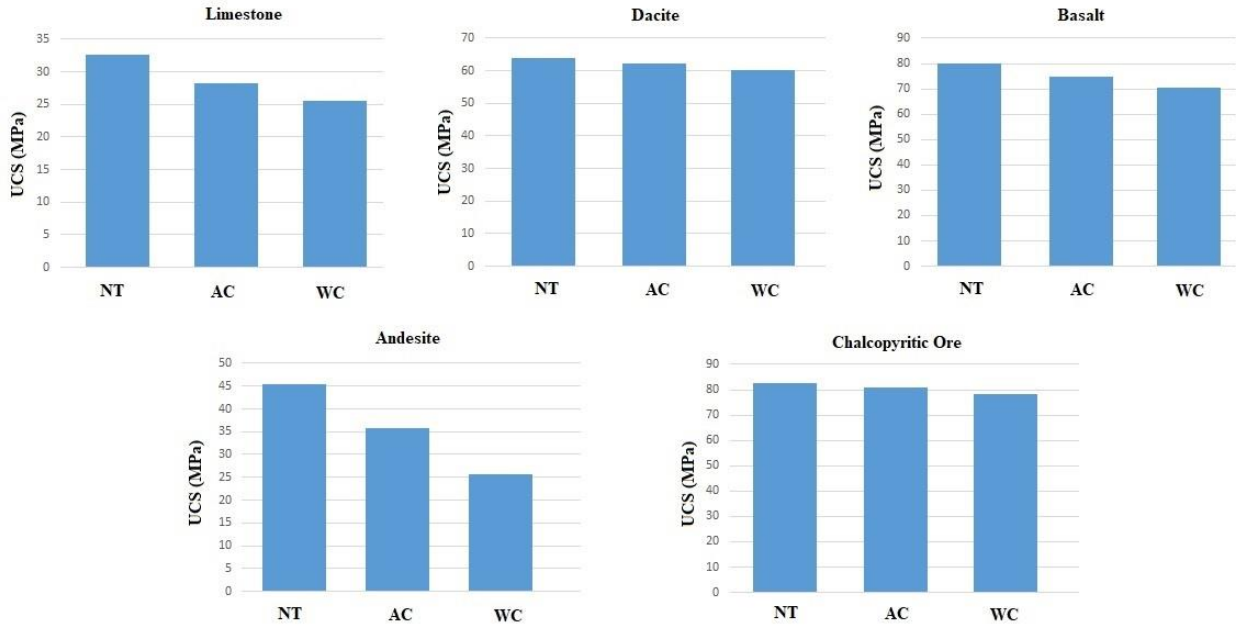


Figure 8: Change in uniaxial compressive strength values under different cooling conditions (NT: No thermal process, AC: Cooling in air, WC: Cooling in water)



#### 4. Discussions

Obtaining increase in porosity and decrease in strength values with increasing temperature and thermal cycle number is parallel with results of other relevant studies carried out by different researchers (Zhang et al. 2018; Kumari et al. 2017; Huang and Xia 2015; Keshavarz et al. 2010). Thermal stresses made increases in the porosity due to the heating and cooling strains. In this respect, mineral contacts are critical for new crack occurrence due to the thermal cycles (Yang et al. 2017; Sun et al. 2016; Mahmutoglu 2017; Mahmutoglu 1998; Mahmutoglu 2006). Especially, thermal strains under high temperature variations are notable for strength losses.

As confirmed by the results of this study, cooling strain values decrease with the increase in thermal cycle number under a same temperature change. This situation verified that the thermal strain capability decreases with a notable increase in porosity resulting from micro-cracking and the start of weathering (Papitha et al. 2013; Lin 2002). As it was seen from andesite specimens, thermal strain limits can also decrease despite the increase in temperature of ongoing thermal cycle. This situation was assessed to be due to the high physical weathering problem of the andesite material. The initial microcracks and high porosity of the andesite material tested within this study should be taken in consideration for having the early physical weathering problem (Sousa et al. 2005; Komurlu and Kesimal 2015).

Some limestone specimens had narrow visible cracks after the fifth cycle. On the other hand, the andesite specimens had started to have cooling cracks after the third step heating from 20 °C to 220 °C and significantly started to be disintegrated at the end of the last and fifth cycle (Figure 6). The cooling environment is truly determinative for the start of cracking and having disintegration (Cai et al. 2015; Kumari et al. 2017; Zhang et al. 2015). Heat capacity of the cooling environment varies the energy transfer amount during the specimen cooling process. As an example, specimens have faster energy loss and cooling for a unit temperature change in water rather than air. Therefore, the specimens cooled in water had less-resistivity against a temperature change in comparison to those cooling in air (Zhao et al. 2018; Brotons et al. 2013).

Because cooling duration increases with an increase in the heat capacity of rock materials, significant variations in the cooling times of different rock materials were obtained in this study. It was seen from limestone and andesite specimens after fourth and fifth thermal cycles that cooling durations obviously decrease as a result of weathering cracks and increase in porosity (Lafdi et al. 2007; Sundarram and Li 2014; Wang et al. 2009).

With an increase in the heat capacity of the cooling environment, durations for a temperature change of rock specimens and completion of the cooling strain decreases. Use of some other cooling liquids with higher heat capacities like varying oil types, gels or specific water based solutions is an open topic for investigating a stronger thermal shock effect on rock materials than cooling in tap water. As another example, ancient miners were using vinegar to make shock cooling by quenching and blowing out fire on rock masses since they found vinegar effective to enable excavation easier due to making cracks (Sanström 1963; West 1988; Komurlu and Kesimal 2013). Freezing temperature of a liquid material has also a significant importance to select a proper environment making a rapid cooling with a good thermal shock.

It was determined that strain levels under thermal stresses can be much higher than those resulting from mechanical stresses under the room temperature. Limit of change in temperature to have cooling strain without cracking is highly restricted in case of a rapid shortening process due to the cooling in an environment with high heat capacity. Because thermal deformation modulus and heat capacities of different minerals of a rock material differ, the mineral contacts are generally the weakest locations in terms of crack initiation and propagation of existing cracks.

As one of the important outcomes of this study, cooling strains under increasing thermal cycle number were found to decrease after a critical cycle. This situation is suggested to be called as cooling strain capacity loss by the author. Definition of cooling strain capacity loss is having a decrease in the cooling strain in comparison with that in previous thermal cycle, under a same or increasing change in temperature. Normally, thermal strain is expected to increase with an increase in temperature change in ongoing thermal cycles. However, it decreases for rock materials with the case of cooling strain capacity loss. This situation can be stated to result from weakening due to the thermal fatigue (Kim et al. 2014; Hall 1999; Vargas et al. 2013). It was determined from this study that rock materials with cooling strain capacity losses had also higher strength losses in comparison with those having no cooling strain capacity loss problem.

It should be noted herein that the cooling strain capacity loss problem and the issue of cooling duration decrease due to a substantial increase in porosity were observed together as seen from fourth and fifth cycles of andesite and limestone specimens. It can be assessed in accordance with the findings of this study that the cooling strain capacity loss and decrease of cooling duration under a same change in temperature indicate a substantial strength loss of rock materials. The critical thermal cycle number and heating temperature for having cooling strain capacity loss resulting from micro/macro cracking and a considerable increase in the porosity of cooled specimens in consequence of the thermal fatigue vary in accordance with rock material type. It was seen from this study that rock materials with a poor thermal durability and early cooling strain capacity loss problem have visible crack occurrences, disintegration and relatively high strength loss values. For instance, andesite specimens which have a high cooling strain capacity loss were found to have major disintegration, whereas hard chalcopyrite from Akarsen copper mine had only some microcracks which are invisible by naked eye. In case of cooling in water, the strength loss of andesite and chalcopyrite specimens were 44% and 5% after the end of thermal cycles, respectively.



## 5. Conclusion

The heating and cooling cycles made notable strength losses and changes in physical properties, depending on rock type and cooling environment. Especially, cooling in water condition made considerable strength losses for all specimens, rather than cooling in air. The rocks with high strength loss values were found to have also high cooling strain capacity losses in comparison with those having less strength loss values. Therefore, cooling strain capacity loss was assessed to be an indicator of the strength loss. The findings from this experimental study are aimed to contribute to new studies on development of a new method for evaluation of thermal change effect on strength loss property of rock materials. In a study of improving a test to evaluate and classify the thermal change resistance of rock materials, it is required to determine the methodological details such as specimen size and geometry, thermal cycle number, heating temperature, cooling material and its temperature. It is also necessary in thermal resistance classification to establish a methodology to rate cooling cracks which can be classified into varying groups like narrow cracks, visible major cracks, surface disintegration and major disintegration.

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