# Driving Force and Thermal Activation Process in PWSCC (primary water stress corrosion cracking) in Alloy 600

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# 1. Introduction

PWR (pressurized water reactor) using Alloy 600 steam generator (SG) tubes and weld 182 dissimilar welds (dissimilar weld metal) suffered damage due to primary water stress corrosion cracking (PWSCC). This phenomenon has been studied for a long time in terms of corrosion since it was first reported in 1957 but is not fully understood[1]. Corrosion in the Alloy 600's PWSCC does not at least explain the following facts; (a) what is the nature of the thermal activation process, which shortens the PWSCC initiation time when operating temperatures increase, (b) what is the nature of the activation energy (Q=185-210 kJ/mol), (c) what is the driving force of the PWSCC, and (d) why PWSCC sensitivity depends on the cooling rate from high temperature mill anneal(HTMA).

Although the PWSCC phenomenon was not fully understood until about 2010, from an economic point of view, PWR suffering PWSCC damage through engineering judgment has been allowed in commercial operation for decades. In order to overcome this limitation of the Alloy 600, the steam generator material in PWR has now been replaced by Alloy 690, and Weld 182 has been replaced by Weld 152.

A PWSCC mechanism based on an ordering reaction has been proposed to overcome this limitation [2,3]. Based on this new PWSCC mechanism, it is advantageous to reduce the cooling rate at high temperatures. And it seems best to apply post-heat treatment to cold working or welding areas. However, it is not clear whether the PWSCC of the Alloy 600 can be fundamentally prevented.

Attempts to explain PWSCC in terms of corrosion are still ongoing [1]. Corrosion is a term that comprehensively refers to the process by which a metal becomes an oxide in a more stable state. In nature, one of the most stable state of metals is oxides. Since the metal used by mankind is returned to metal by reducing ore, thus, the metal is unstable and tries to return to its oxide form in nature. Corrosion is that the metal tries to move to a more stable state, so corrosion is inevitable like gravity. However, although corrosion is inevitable, it seems illogical that the PWSCC of Alloy 600 is controlled only by corrosion in aqueous solutions.

Corrosion is a surface reaction in contact with oxygen or solution[4]. One such form is largely anodic dissolution in which a metal is dissolved in the form of ions. Anodic dissolution occurs when exposed to a corrosive solution such as an aqueous solution. A typical example of anodic dissolution is a plating process. For example, when copper is plated on iron, the copper anode dissolves and supplies copper ions to the plating solution, and the copper adheres to the iron surface of the opposite cathode and is plated. The phenomenon in which the anode is dissolved with a plating solution is called anodic dissolution. Since the anode is dissolved into ions, anodic dissolution can be said to be a kind of corrosion.

Another form of corrosion is when oxygen adheres to the surface of the metal to make an oxide and/or corrosion product. The surface oxide attached in this way also serves to protect the material by preventing the base metal from being exposed to the corrosion environment. This state is called passivity, and it is a term that refers to a state in which the initial corrosion action almost stops and corrosion does not proceed anymore. This effect appears in many metals or alloys and increases corrosion resistance. In other words, this type of corrosion is very different from anode dissolution in that it creates corrosion products on the metal surface. In the anodic dissolution process, the metal dissolves and no corrosion products are generated at all, but when the corrosion product is formed on the surface, the metal surface is hardly directly exposed to the corrosion solution, resulting in an antioxidant effect. Damage to inter-granular stress corrosion cracking (IGSCC) in alloys having a composition similar to that of the Alloy 600 occurs not in aqueous solutions but also in argon (Ar) environments [5]. This result shows that intergranular (IG) fracture occurs regardless of corrosion. To be more precise, the grain boundary (GB) fracture in Ar is irrelevant to corrosion. Therefore, the PWSCC of the Alloy 600 strongly suggests that there is a driving force to form the GB crack regardless of

corrosion. However, as described above, anodic dissolution or oxide film formation does not explain the generation of driving force that opens the GB. The reason is that although corrosion is a surface reaction, the exposed environment in which the material changes is a phenomenon that occurs in the entire material. The PWSCC of Alloy 600 commonly occurs in area in contact with high-pressure primary water. However, even in the process where the anodic dissolution dominates, the GB may dissolve a little faster, but the anode itself cannot provide the driving force for total GB fracture.

Let's think about whether IG fracture or PWSCC is a surface problem or a bulk problem. PWSCC occurs when the entire material is exposed. That is, in the Alloy 600, the entire material causes any changes in the PWR environment. However, if we look at the PWSCC phenomenon occurring in the reactor environment only from the viewpoint of corrosion, there is a limitation that only changes in the material surface are considered. Corrosion occurring on the surface of a material cannot explain damage occurring throughout the material. In other words, just because surface corrosion is inevitable does not mean that surface corrosion is caused by changes in the bulk material. Oxidation of the material surface could have some effect on component damage. However, the view that corrosion causes GB damage is narrow-minded.

As presented above, the reason for not being able to explain (a)-(d) seems to be that only the surface reaction called corrosion was considered without considering changes in the entire material in the material exposure environment. There is a need for a paradigm shift in that changes to the entire material will have a greater impact if the entire material is exposed. The surface is only a part of the material. In other words, the claim that the bulk of the part is damaged due to corrosion where part of the material oxidizes is contradictory.

This paper attempted to explain the PWSCC phenomenon that occurs in Alloy 600 using a mechanism based on ordering reactions [2,3]. The PWSCC mechanism based on the regularization mechanism can explain (a)-(d), which cannot be explained by corrosion phenomena, as listed above.

## 2. Driving force for PWSCC and detection of

### cracking during ordering reaction in Alloy 600

The IG fracture of the Alloy 600 occurs in the form of fracture of GB. As cited above [5], the IG fracture of the Alloy 600 occurs regardless of corrosion. This means that the force to open the GB acts during IG fracture. The PWSCC mechanisms [2, 3] based on the ordering reaction can explain well how the driving force to open the GB occurs in the Alloy 600.

If the Alloy 600 is maintained at a high temperature, the

atomic arrangement becomes irregular, increasing the entropy and disrupting the atomic arrangement, resulting in a three-dimensional expansion of the lattice. Exposure of the Alloy 600 in this state to a temperature of about 330°C changes the atomic arrangement to a stable one through the diffusion process, and the lattice contracts at the same time, providing a driving force to

open the GB as the grain contracts.

Figure 1 is a differential scanning calorimeter (DSC) analysis of the WQ Alloy 600 specimen after maintaining the Alloy 600 containing 0.009% carbon at 1,095°C. FC alloy 600 is in a state of DSC analysis of the WQ specimen and cooling it in the device. This experimental result is to compare the difference in cooling rate in the same specimen.



Figure 1. Comparison of thermal analysis curves through differential scanning calorimeter (DSC) for WQ and FC Alloy 600.



Figure 2. Comparison of anisotropic lattice contraction in (111), (200), and (220) planes measured by neutron diffraction in HANARO in KAERI according to ordering treatment at 400°C in WQ specimens.

As shown in Figure 1, only WQ Alloy 600 shows an exothermic reaction, but FC does not. In other words, the exothermic reaction occurs because it is due to fast cooling by water quenched(WQ) from high temperature.

When a material in this state is rapidly cooled by the WQ method, the atoms are remained in an excited state and the entropy of the material increases. When the material in this state is rapidly cooled by the WQ method, the atom is cooled to an excited state, leaving the atomic arrangement in the excited state to room temperature and thermal energy. The remaining thermal energy is due to the unstable arrangement of atomic arrangements, so entropy remains essentially. If the change in the average lattice distance is measured while ordering the Alloy 600 in this state at 400°C, the state with high entropy becomes a three-dimensional expanded state. If the material in this state is maintained at 450°C or less where the exothermic reaction occurs, the entropy decreases and the average lattice distance contracts.

Figure 2 investigates the lattice contraction behavior according to the ordering treatment time at 400°C for up to 5,000 hours. This result was confirmed by measuring with a high resolution powder diffraction (HRPD) device installed in the reactor as one of the Korea Atomic Energy Research Institute.

Figure 2 shows the lattice distance change rate of the WQ Alloy 600 at 400°C according to the ordering treatment time. It may be seen that a material having high entropy by WQ treatment contracts during ordering treatment and this means that Alloy 600 was expanded in three dimensions.

Figure 3 shows the average lattice distance change rate of (111) plane according to the ordering treatment time at 400°C after changing the cooling rate by WQ, air cooling (AC) and furnace cooling (FC) treatment after solution anneal (SA) treatment. The WQ specimen is saturated with a contraction rate of about 0.035% and rapidly lattice contraction occurs up to 500 hours. The WQ specimen with the largest cooling rate shows a contraction rate about twice as large as that of AC or FC. This is a clue to explain the effect of the cooling rate in the thermal treatment for the Alloy 600.



Figure 3. Comparison of lattice change rates of (111) plane according to cooling rate.

For materials with high entropy by WQ treatment, the entropy decreases and stabilizes as the atomic arrangement is aligned. These materials are diffused as they rise to a high temperature such as the operating temperature of the reactor, and the atomic arrangement is aligned and the entropy decreases. The important point here is that as the entropy decreases, the expanded lattice contracts as it changes to a stable state. In the Alloy 600 made up of atoms, the length of the entire material decreases as the distance between atoms becomes closer, and this crystal contraction creates a tensile (+) stress in the GB by itself inside the material. This is the driving force of IG fracture and/or PWSCC in Alloy 600.

The type of ordering involved in the short range ordering (SRO) reaction varies depending on the alloying elements. Alloy 600 is an alloy having a composition of 72Ni-16Cr-9Fe. When these three elements are mixed, a Ni2Cr phase may be formed between Ni2Cr, NiFe, NiFe, NiFe3, and Fe-Cr between Ni3Fe, NiFe, and Fe-Cr. Therefore, since the bonds between specific atoms are stable, forming stable bonds in a size range of several tens of A (angstrom) is called SRO reaction. When the SRO reaction occurs in this way, it is due to the ordering of the heterogeneous atomic arrangement, and thus the mixing entropy component decreases. Atomic configuration entropy is influenced by how the types of atoms are arranged, but is formed by disrupting the direction of the atomic arrangement in a pure metal. The configuration entropy component is much larger than mixing entropy and is important because it occurs in all materials.

Considering only the mixing entropy of the material, it can be said that the SRO reaction meant that the mixed entropy component was lowered. However, the SRO reaction at Alloy 600 takes into account the configuration entropy and the mixing entropy. Therefore, it is more comprehensive to express the SRO reaction of a material as entropy reduction. This is because the exothermic reaction caused by entropy reduction in WQ materials cannot distinguish between the configuration and the mixing component. If this fact is premised, the expression that the ordering occurs by the SRO reaction and the expression that the entropy of the configuration and mixing components decreases have the same meaning. Driving force for PWSCC is developed by a decrease in entropy remained by WQ in the material. Materials cooled faster have a larger entropy. The driving force generated by entropy reduction due to the SRO reaction increases as the entropy decrease increases. Therefore, materials with a high cooling rate create a greater driving force. As shown in Table 1, the PWSCC initiation experimental results according to the cooling rate show that WQ is more vulnerable than AC or FC treatment after WQ at high temperature. It can be well confirmed that this is related to the entropy remaining in the Alloy 600 by WQ, as shown in Figure

1.

The root cause of the GB of the Alloy 600 is due to the lattice contraction due to entropy reduction. As can be seen in Figures 2 and 3, when the WQ Alloy 600 is subjected to ordering at 400°C for about 2,000 hours, the (111) surface shrinks by about 0.035%. Since this contraction rate is the same as the deformation ( $\epsilon$ ) by the regularization treatment, it can be converted into stress by Hook's law ( $\sigma$ =E $\epsilon$ ). The WQ Alloy 600 generates a tensile stress of at least 0.035% x 220 GPa = 77MPa due to the ordering reaction. Due to the decrease in entropy due to the SRO reaction, the Alloy 600 generates a tensile stress of about 77 MPa and becomes a force to open the GB, thereby serving as a driving force for IG fracture.

The stress of 77 MPa generated by lattice contraction is less than the stress required to reproduce PWSCC in a low strain rate test (SSRT) experiment, but it is sufficient to open the GB. The stress applied to reproduce PWSCC causes deformation during the experiment, so it is different from IG fracture occurring in PWR by lattice contraction. In the WQ Alloy 600, the saturated contraction rate of the (111) plane by the normalization reaction at 400°C is about 0.035%. If the grain size of the Alloy 600 is 100µm, crystal contraction of about 35 nm(=100µm x 0.035%) occurs in a direction perpendicular to the (111) plane. This can be mitigated or amplified by surrounding crystals. When the GB is opened, the atomic bond or GB may be broken even if only one atomic layer is reduced. In this way, lattice contraction due to entropy reduction becomes the driving force of PWSCC. Figure 4 shows GB cracking by lattice contraction.

a) as-treated direction of crystal b) nano crack formed at high angle boundary) (nano crack formed at high angle boundary) crack

Fig. 4. Schematic illustration of the nano-scale intergranular cracks formation due to the anisotropic contraction of a crystal.

In order to reproduce the IG fracture of the Alloy 600, the tensile strain applied in the SSRT experiment near 330°C increases the entropy by itself. Plastic or elastic deformation is a process of increasing the entropy by itself because the atomic arrangement is disturbed. The SSRT experiment continuously increases entropy through tensile strain, and the Alloy 600 contracts the lattice contraction by atomic diffusion process at 330°C, so IG fracture is quickly reproduced. That is, even in the SSRT experiment, the driving force of IG fracture is provided by lattice contraction due to entropy reduction. The SSRT experiment continuously supplies entropy to the specimen through deformation, but for the reactor part, there is a difference in that only the entropy left during the PWR manufacturing process contributes to IG fracture.

Figure 5 shows the acoustic emission (AE) counts generated during the process of installing and heating a WQ Alloy 600 specimen in a device that can check AE. AE signals are observed at the temperature where an exothermic reaction occurs between 450-600°C. The red curve shows lower Cp (specific heat) value compare to the blue curve analyzed by DSC in Figure 5. This is a exothermic reaction due to release in entropy in WQ Alloy 600.

AE is an elastic wave that appears when the elastic strain energy stored until then is released when a solid is deformed or fractured. In this experiment, only heating was performed while the specimen was hung on a cantilever. Therefore, the AE signal observed in this experiment does not appear due to deformation. This AE signal appears from a broken bonding of atom at grain boundaries. The fact that the AE signal appears in the process of decreasing the entropy of WQ Alloy 600 means that GB crack and/or fracture is occurring as atomic bonds are broken due to lattice contraction. Therefore, lattice contraction caused by a decrease in entropy caused by atomic diffusion provides the driving force for IG destruction.



Figure 5. Comparison of Cp (specific heat) and acoustic emissions (AE) during heating of WQ Alloy 600.

# 3. The nature of the thermal activation process and its activation energy for PWSCC

IG fracture in PWR is sometimes divided into initiation and propagation stages. Experimentally, PWSCC reproduction experiments measure initiation or initiation time using specimens such as U-bend, and PWSCC

propagation speed is measured using CT (compact tension) specimens under load. In artificial reproduction experiments, initiation and propagation can be separated in the laboratory test, but it is difficult to distinguish between initiation and propagation stages in PWSCC that actually occurs in nuclear reactor components. The reason for this is that the width of the crack in PWSCC is so narrow that it is not well detected by nondestructive testing, and it is often confirmed as the primary water in the reactor leaks to the outside. However, the concept of initiation and propagation emerged because it takes time for partially advanced defects before IG fracture penetrates logically. In addition, since it can be very important engineering to determine when IG defects in non-penetrating parts penetrate, it is necessary to quantify the propagation rate. The thermal activation process refers to a phenomenon in which the time when PWSCC initiation occurs in a PWR reactor components decrease as the operating temperature increases. For this reason, it is an empirical fact that PWSCC initiation of reactor components to which Weld 182 is applied occurs fastest at the highest operating temperature, such as a pressurizer. When the operating temperature increases like this, the time to damage is shortened due to the diffusion rate that increases as the operating temperature increases. A typical thermal activation process is the diffusion process of atoms.

Solution anneal (SA) treatment is applied in order to remove carbide precipitated in Alloy 600. This temperature depends on the amount of carbon concentration. When maintained at such a high temperature, the atomic arrangement of the Alloy 600 becomes disordered and excited state. When this is subjected to WQ treatment, the disordered and excited atomic arrangement remains at room temperature. This state has high entropy and is unstable. Thus, when it reaches at high temperature at which atomic diffusion is possible, it tries to become a stable atomic arrangement through diffusion. That is, the driving force of PWSCC occurs in the process of decreasing entropy through the SRO reaction, and this reaction is dominated by atomic diffusion. This is the nature of the thermal activation process in the phenomenon of IG fracture and/or PWSCC in Alloy 600.

The thermal activation energy for this SRO reaction is measured as Q=190 kJ/mol when the WQ material is thermally analyzed bt DSC in WQ Alloy 600 at 1095°C containing 0.009% carbon [5, 6]. This value agrees well with the Q=180-210 kJ/mol for PWSCC reported by the French EDF. On the other hands, the activation energy for the SRO reaction in WQ Alloy 690 is measured as Q=250 kJ/mol.

WQ Alloy 600, where carbides are not empirically precipitated at the GB, is more vulnerable than thermal treatment (TT) Alloy 600 where GB carbides are precipitated. Table 1 shows that the WQ Alloy 600 is the most vulnerable in the PWSCC initiation experiment

using the U-bend specimen. This result is a round robin experiment conducted in international collaboration around in 2000 to understand the effect of cooling rate on PWSCC initiation. This experiment confirms whether PWSCC is initiated by changing the cooling rate after solution anneal treatment by using the WQ, AC, and FC method and making a U-bend by putting 24,000 hours at 332°C in the primary water environment. In the state of WQ, PWSCC occurred 100% but no PWSCC occurred at all for AC and FC. This experiment shows how important the cooling rate of the Alloy 600 is after SA treatment.

Comparing the GB Cr concentrations of WQ and TT after SA treatment, the GB Cr concentration of WQ Alloy 600 is little different from the matrix, but TT Alloy 600 has a Cr deficiency of about a few percent as GB carbides are formed. However, the fact that WQ Alloy 600 without Cr deficiency is more vulnerable to PWSCC than TT shows that PWSCC is not dominated by corrosion.

Table 1. PWSCC initiation experiment using U-bend (International round robin test using U-bend through ICG-EAC meeting in 2000).

# SCC tests at 332°C for ~24,000h in simulated primary water

Solution Temp.	Cooling Rate	Number of Cracked Samples
1025ºC for 1h	WQ (water-quenching)	<mark>10</mark> /10
	AC (air cooling)	0/10
	FC (furnace cooling)	0/10

# 4. Comparison of susceptibility on PWSCC in Alloy 600 and Alloy 690

Alloy 600, which was used as a nuclear reactor steam generator tube, has been replaced by Alloy 690 due to its high PWSCC sensitivity. This is because Alloy 690 was difficult to reproduce PWSCC in the laboratory. Alloy 600 contains about 16% of chromium (Cr) as an alloy element, while Alloy 690 contains about 30% of chromium (Cr), so its composition is different. From a corrosion perspective, there is an implicit consensus that the PWSCC phenomenon is caused by corrosion, with the explanation that Alloy 690, a material with a high chromium content, has high corrosion resistance from the viewpoint of corrosion.

As shown in Figure 6, WQ Alloy 600 and Alloy 690 from 1100°C clearly cause exothermic reactions, but the exothermic temperatures are different. The essence of

this exothermic reaction is that the atoms in the excited state during the solution annealing process are rapidly cooled and cooled to the excited atomic arrangement state, and the state with increased entropy returns to the stable arrangement, resulting in heat released. Since this occurs through atomic diffusion that occurs in a short range, it can be called a SRO reaction. The process of stabilizing the atomic arrangement from an excited state can be expressed as an SRO reaction or a decrease in entropy.



Figure 6. Comparison of Cp (specific heat) curves of WQ Alloy 600 and WQ Alloy 690.

Table 2. Comparisons of Arrhenius rate parameter of Alloy 600 and of Alloy 690 at 330°C.

Alloy	Arrhenius parameter at 330°C
Alloy 600 of Exp(- Q/RT), (Q=190kJ/mol)	3.5 x 10 <sup>-17</sup>
Alloy 690 of Exp(- Q/RT), (Q=250kJ/mol)	2.2 x 10 <sup>-22</sup>
Ratio of Arrhenius parameter at 330°C (Alloy 690/Alloy 600)	6.3 x 10 <sup>-06</sup>
1/(Ratio of Arrhenius parameter at 330°C)	1.57 x 10 <sup>5</sup>

As shown in Figure 6, thermal analysis of Alloy 600 shows the maximum heat generation peak temperature (Tp) at around 520°C, and Alloy 690 shows Tp at 600°C. This means that when the reactor operating temperature is 330°C, Alloy 600 and Alloy 690 have a difference from Tp of 190°C and 270°C, respectively. As the SRO reaction occurs in Alloy 600 and 690, the reaction that reduces entropy occurs through diffusion. The rate of this diffusion reaction follows the Arrhenius rate equation: rate  $\propto \text{Exp}(-\text{Q/RT})$ . Where Q is the activation energy, R is the gas constant, and T is the absolute temperature. The activation energy for the SRO reaction is about 190 kJ/mol and 250 kJ/mol for Alloy 600 and 690, respectively.

Therefore, atomic diffusion in Alloy 690 becomes much more difficult, whose temperature differs by more than 270°C from its operating temperature, to cause an exothermic reaction. In other words, Alloy 690 has a slower entropy reduction rate than Alloy 600. The rate of SRO response of Alloy 690 is much slower than that of Alloy 600.

When the reactor operating temperature is 330°C, the diffusion rate (Arrhenius rate) of the SRO reaction of Alloy 600 and Alloy 690 is compared as shown in Table 2. The ratio of diffusion rate of Alloy 690 is lower than that of Alloy 600, about 6.3x10<sup>-6</sup>. The SRO reaction that would occur in one year in Alloy 600 takes approximately 157,000 years ((-1/6.3x10<sup>-6</sup>)=157,000) in Alloy 690 at 330°C. The rate of entropy reduction in Alloy 690 is approximately 157,000 times slower than in Alloy 600. The reason why the PWSCC resistance of Alloy 690 is higher than that of Alloy 600 is not because the high Cr concentration slows down the corrosion rate, but because the generation rate of PWSCC driving force is slow in a nuclear reactor environment at 330°C. In other words, the driving force of PWSCC is not generated by corrosion, but by entropy reduction due to the SRO reaction. The PWSCC resistance of Alloy 690 can be explained independently of the corrosion process.

The ordering behavior of Alloy 690 and Alloy 600 is slightly different. The composition of Alloy 690 is 60Ni-30Cr-9Fe, while Alloy 600 has a slightly different composition of 72Ni-16Cr-9Fe. Alloy 690 has Ni:Cr=2:1 excluding Fe, which is the same as the ordering composition of Ni<sub>2</sub>Cr. Accordingly, Alloy 690 may form a long range order (LRO) Ni<sub>2</sub>Cr phase through ordering processing, while Alloy 600 does not create an LRO.

In this process, the grain boundary tensile stress of Alloy 600 is amplified throughout whole grain due to entropy reduction. This causes grain boundary crack. On the other hand, Alloy 690 is thought to not amplify the tensile stress formed at grain boundaries by creating fine LRO according to the relationship of 6 variants within grain, like orthohombic Ni2Cr. This is assumed to alleviate the role of tensile stress at grain boundary in spreading grain and/or grain boundaries. Alloy 690 appears to be PWSCC resistant because the ordering rate is slow than that of Alloy 600 and the grain boundary tensile stress formed during LRO process is not amplified.

### 5. Conclusion

(1) The driving force for the PWSCC of Alloy 600 is provided by lattice contraction due to the entropy reduction process by the SRO reaction. The thermal activation process for the initiation of PWSCC in Alloy 600 is governed by atomic diffusion as the entropy reduction process by the SRO reaction. (2) In Alloy 600, the nature of the thermal activation process of the PWSCC is for the atomic diffusion reaction caused by entropy reduction.

(3) The susceptibility of PWSCC in the Alloy 600 depends on the cooling rate after SA treatment. As the cooling rate increases, the disordering of the atomic arrangement increases further, so the amount of residual entropy increases, and the lattice contraction rate due to entropy reduction increases. This is why the Alloy 600, which has a high cooling rate, is more vulnerable.

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