

## Work Hardening and Softening of 4-6N Aluminum in the Processing of Cold Rolling and Heat Treatments

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Work-hardening is a well-known phenomenon in metallic materials that cold working raises the hardness of materials. However, the work-softening phenomenon in which hardness is reduced by working is also observed in metals and alloys. The investigation of details of work-softening has remained underdeveloped so far. There has been no interpretation established to deal with work-hardening and softening simultaneously, despite both hardening and softening occur in similar materials. In this study, we have focused on the relation between hardness and purity, processing conditions in aluminum. The present study has concluded that the work-hardening and softening phenomena appearing in aluminum depend on the purity level of materials, and the phenomena cannot be attributed to the behavior of dislocations.

**Keywords:** work-hardening, work-softening, purity of metal, aluminum

### 1. Introduction

Although metals and alloys are often hardened by cold working, the hardness decreases and the material becomes softened when high purity materials are strongly wrought. Phenomena opposite to work-hardening have been reported in a number of metals and alloys, and this type of phenomena can literally be called "work-softening". It is 1950s that such work-softening was found in high purity aluminum. In addition to pure metals, it was also found that work-softening occurs in an annealed Al-Fe alloy containing 0.5~1.5mass% of iron (cf. Fig.1). Those phenomena have been interpreted by dynamic recovery or dynamic recrystallization [1-4]. But there are questions what factor dominates the stability of dislocations and their accumulation, and what is the critical condition to recovery and recrystallization [5]. In this study, we have focused on the influence of the purity of aluminum, and considered the work-hardening and work-softening phenomena.

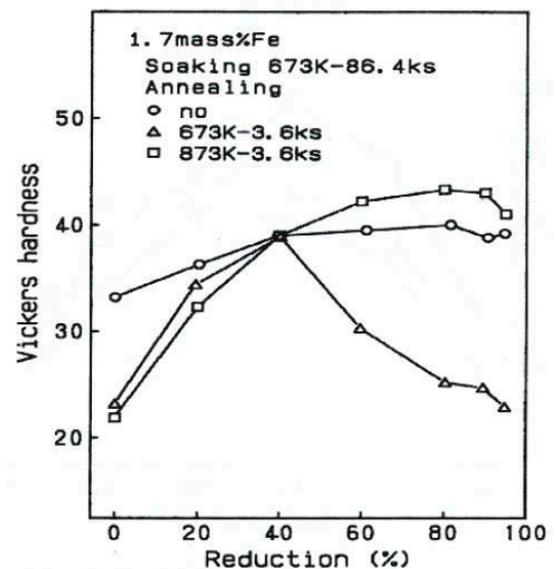


Fig.1. Vickers hardness of Al-Fe alloy

### 2. Experimental

We prepared four pure aluminum specimens with different purity levels from 3N to 6N. To examine the relation between the hardness and processing condition, all specimens with different purities were dealt with same experimental procedure in this study. The specimens were annealed at 573K, 673K, and 773K for 10min and 100min. The cold rolling was carried out at room temperature by a mill with reduction ratios, 30%, 60% and 90%, respectively, either before or after heat treatments, to introduce plastic deformation into the specimens. On the deformation process, the rolling was performed with

care for avoiding the temperature rise of the specimens. After the processing, we measured the hardness of the rolled specimens using a Shimadzu HMV-2000 Vickers microhardness tester with 0.98N load and holding time of 30sec.

To investigate the relation between the hardness and the microstructure changes after the processing, we carried out the hardness measurements and TEM observations using a HITACHI H-800 TEM operated at 175kV. All TEM images were taken at the region of central part of thickness from the normal direction of specimens, perpendicular to a compression axis.

### 3. Results and Discussion

#### 3.1 HV hardness of Al specimens receiving heat treatment before cold rolling

Figure 2 shows Vickers microhardness on cold rolling. After reduction of 60%, the hardness did not change in a 5N sample, whereas the work hardening occurred in 3N and 4N samples. Work-softening was observed for high purity sample 6N in purity when the material received further rolling. It might be worthy to point out that the hardness of the 6N sample receiving 90% reduction is lower than the initial value of the sample. According to these results, it is concluded that the amount of the impurities remaining in aluminum gives influences to the hardness behaviour of the materials in the deformation process. Namely, work-softening occurs in the purity of 5N~6N but when Al metal contains more impurities than 5N, work-hardening occurred, contrarily.

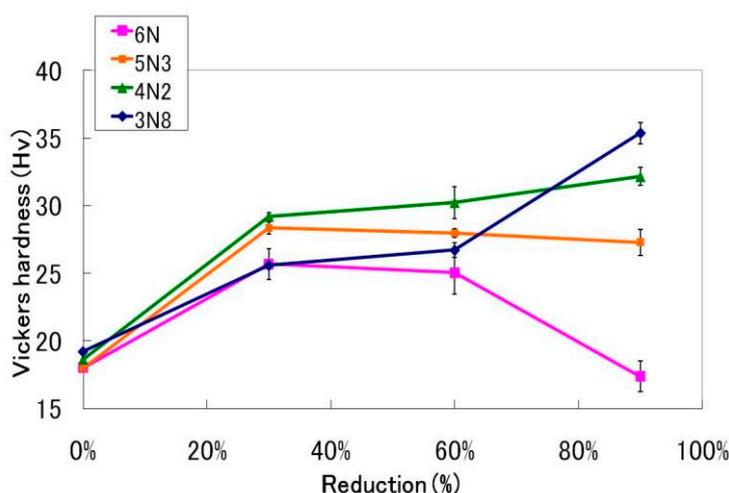


Fig.2. Vickers hardness of pure aluminum without annealing

#### 3.2 Effect of annealing before cold rolling

When metal and alloy sheets are fabricated from ingots, rolling and heat treatments are repeated in the process. We examined the effect of annealing given to a sample prior to cold rolling. Figure 3 and Figure 4 shows the relation of HV and annealing. The influence of annealing time and temperature is not so clearly seen in 6N Al specimen and work-softening occurred, regardless to annealing condition.

However, in 5N Al specimens, the hardness increased with the annealing either at higher temperature or for longer time. Thus, the present results of hardness measurements clearly show that work-hardening and softening depends on purity levels, and that the heat treatment makes the impurity level of specimens enhanced. The latter effect seems unexpected. A probable interpretation to the present results is that the impurity atoms localized in the specimens are dispersed in Al matrices due to heat treatments. The present results also revealed that heat treatment can bring both a positive and a negative effects into materials, as far as the impurity level of matrix is concerned. That is, heat treatment accelerates the diffusion of impurity atoms and reduces dislocations at certain temperatures.

But the treatment at high temperatures can actually increase the impurity level, and this increase causes the negative effect to successive plastic deformations.

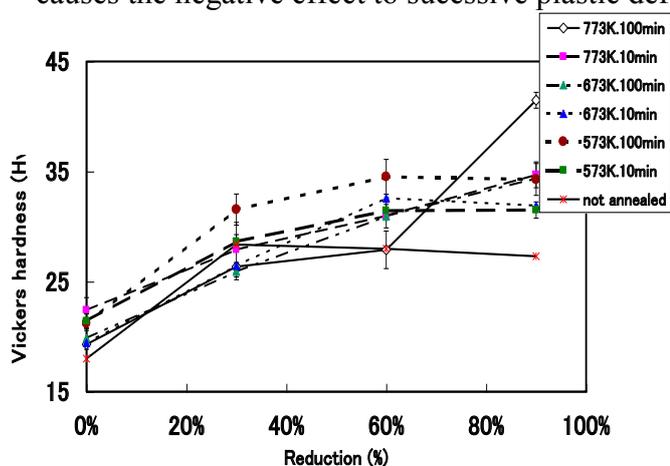


Fig.3. Vickers hardness of 5N pure aluminum annealed after processing.

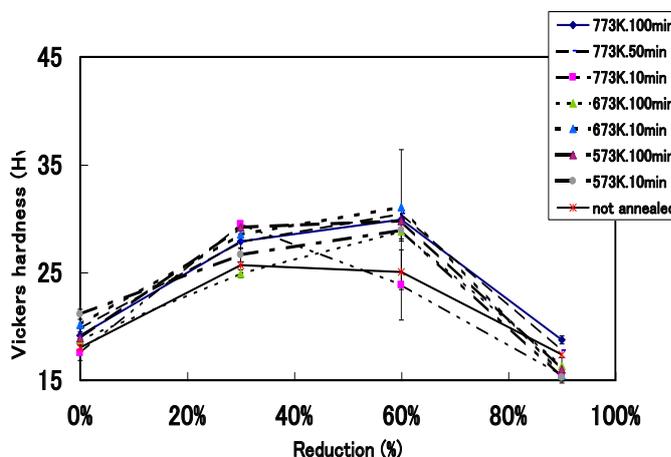


Fig.4. Vickers hardness of 6N pure aluminum annealed after processing.

### 3.3 The HV hardness of Al specimens receiving heat treatments after cold rolling

Figure 5 shows the HV hardness of 5N Al specimens. The hardness was measured after cold rolling and subsequent heat treatments. The annealing at 373K for 100,1000min. hardly affected the hardness, but the annealing reduced the hardness after rolling with reductoin ratio of 90%. It is noted that the hardness without annealing show a small peak at 60% in redction and the peak becomes more sharp after annealing for 5N Al specimen. Figure 6 shows HV hardness of 6N Al receiving similar treatments. Although the hardness again shows the peak at 60% in reduction ratio, the heat treatments gave large effects to reduce the hardness of the 6N Al specimen. Receiving the annealing at 373K for 10min and 100min, the peak position moved to low reduction ratio, and simultaneously hardness itself was remarkably decreased. Comparing Figs.5 and 6, we also notice that the peak separating hardening and softening moves towards lower reduction ratio when the purity of specimen or annealing time is increased. Thus, the purity of metal has a role equivalent to annealing. To this end, it is concluded that the impurity of material dominates the stability of dislocations and the chemical bond nature should be considered when the hardening mechanism is considered.

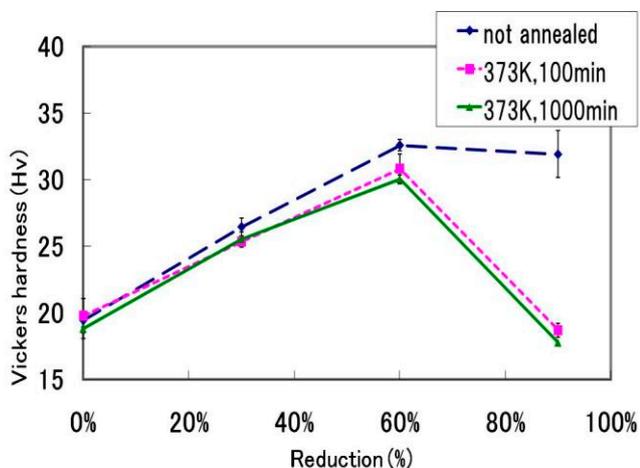


Fig.5. Vickers hardness of 5N aluminum annealed after colled rolling.

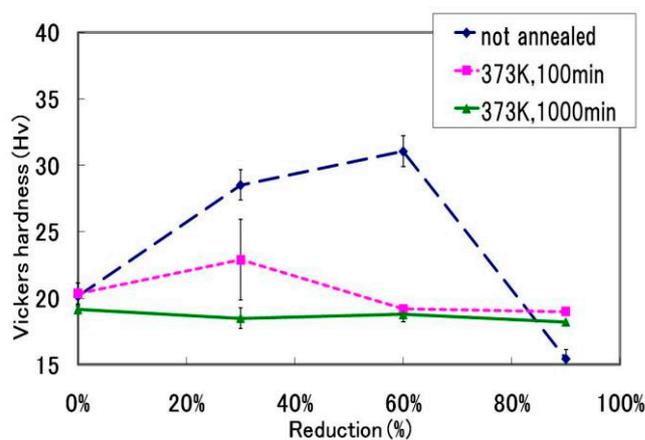


Fig.6. Vickers hardness of 6N pure aluminum annealed after

### 3.4 TEM observations of microstructures occurring during cold rolling in 6N Al specimens

The microstructural evolution of 6N Al specimens were examined by TEM. Figure 7 shows the bright-field TEM images taken at three stages of cold rolling. After annealing the as-received specimen at 773K for 10min, we saw that dislocations sparsely distributed in an aluminum matrix. We also notice that grain sizes are much larger than those observed after rolling of 60%. In the specimen rolled with the reduction ratio of 60%, grain sizes are much smaller than in the initial condition and lots of dislocations were introduced in the specimen. In the specimen rolled down with the reduction ratio of 90%, the tangles of dislocations still remain, but the regions with high density of dislocations are smaller and grain sizes seem larger than the specimen with reduction of 60%. We also find that dislocations are localized in the area, and few dislocations are observed in large grains, at the final stage of cold rolling. This feature is similar to the feature conventionally described as dynamical recrystallization, although the terminology scarcely gives essential information to understand the detail of the phenomenon itself.

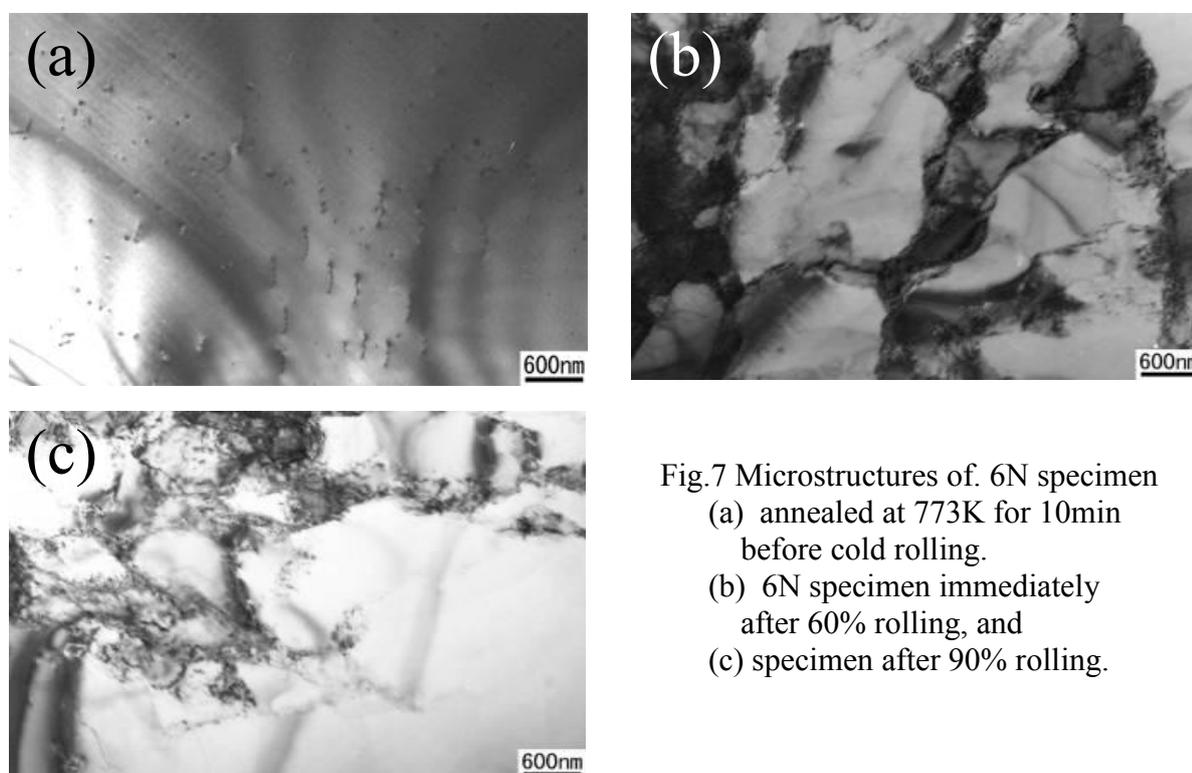


Fig.7 Microstructures of. 6N specimen  
 (a) annealed at 773K for 10min  
 before cold rolling.  
 (b) 6N specimen immediately  
 after 60% rolling, and  
 (c) specimen after 90% rolling.

### 3.5 Influences of the impurities on work-softening

In the above sections, we described the experimental results of the hardness tests and TEM observations. According to those results, we can recognize that the impurities have predominant roles to determine whether hardening/softening occur or not. Although the experimental identification of elements responsible for the phenomena is the most desirable work, the practical approach seems to be a hard task to be performed, due to the concentration levels. Instead of this approach, we considered the influences of two important elements, i.e., Fe and Si atoms on the basis of the extended Hueckel molecular orbital calculations [6]. As is already implied, the impurity atoms modifies the electronic states of Al matrices. When the impurity atoms are dissolved in matrixe, they give influences to the electronic structure. On the contrary, the impurity atoms have no significant influence to the stability of Al matrices, if they are immiscible with the matrices. From this point of view, the solubility of guest atoms can be considered, taking the energetic fluctuation ( $\Delta E$ ) of the states of alloys. Figure 8 shows the calculated fluctuation,  $\Delta E$ , together with cohesive energy of an

Al-Fe alloy. The calculated results show that Fe atoms have a tendency to form an intermetallic compound with an intermediate composition. The fluctuation is not small and subsequently the diffusion seems to occur at 400K which is a practically important temperature. Figure 9 shows the calculated results of an Al-Si alloy. In this case, no significant change in the cohesive energy. The fluctuation does not have an explicit minimum at any composition. Therefore, Si atoms have no tendency to be easily segregated, whereas Fe atoms can be excluded by annealing. Thus, the present EHMO calculations suggest that the work-softening occurs in Al-Fe alloy, but not in Al-Si alloy.

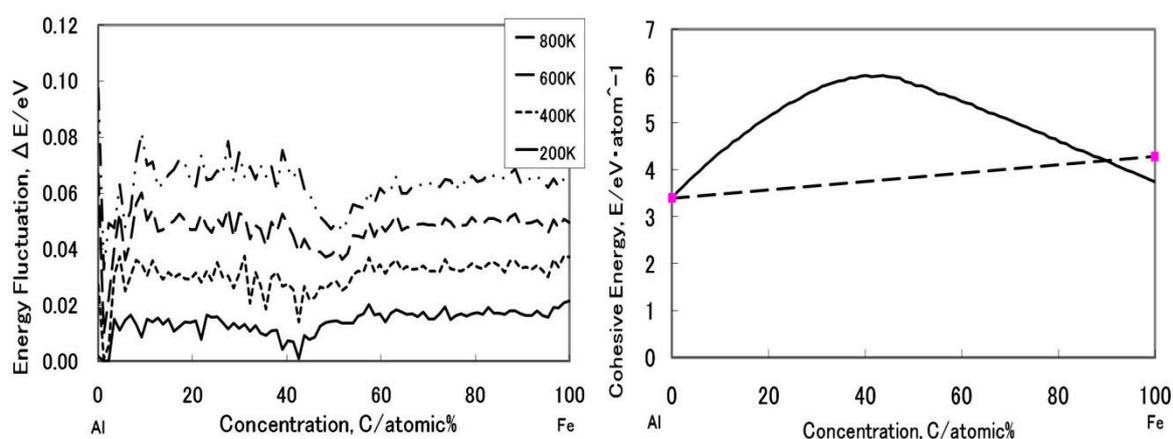


Fig.8. Energy fluctuation (a) and cohesive energy (b) vs.solute concentration in an Al-Fe alloy.

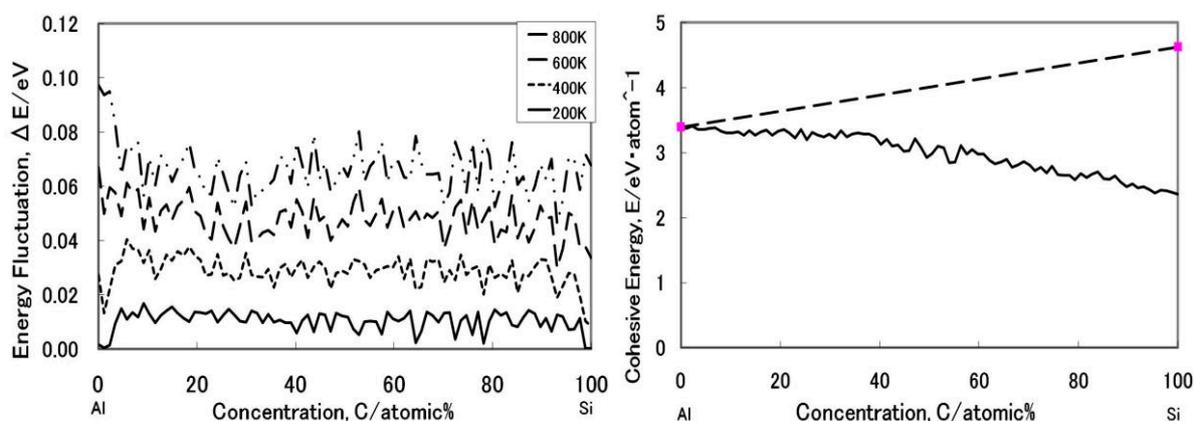


Fig.9. Energy fluctuation (a) cohesive energy (b) vs. solute concentration in an Al-Si alloy.

#### 4. Conclusions

The present study dealt with the work-hardening and work-softening observed in high purity Al. According to the experimental results obtained in this study, it was revealed that the purity level of the materials gives significant influence to the mechanism. Unlike the expectation drawn from the conventional concepts, heat treatments sometimes bring the effect to increase the hardness of materials. An alternative approach based on the chemical bond theory will extend a basic understanding to the properties and the backgrounds of processing consisting of heat treatments and plastic deformations.

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