Re-examination of creep behaviour of high purity aluminium at low temperature

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Abstract. The deformation behaviour of high-purity aluminium at low temperatures was investigated in order to re-examine Ashby-type deformation mechanism map. All specimens with different purities showed significant creep below room temperature. Under the same stress and temperature, the steady-state creep rate increased with increasing purity of the material. They showed stress exponents around 5.0 and apparent activation energies around 20 kJ/mol at temperatures below about 400 K, and 4.0 and 70-80 kJ/mol at temperatures above that temperature. The grain size had no effect in the low temperature region. From the microstructural observation, secondary slip system was observed. These features imply that pure aluminium deforms in the different mode from the ambient temperature creep of h.c.p. metals which has similar activation energy.

1. Introduction
Deformation mechanisms of metals are often plotted in Ashby-type deformation mechanism maps [1]. The map is composed by normalized stress, homologous temperature and strain rate, and the deformation mechanism is given as a function of these parameters. In the map of pure aluminium (Al) drawn by Frost and Ashby [1], creep deformation should proceed even below 0.4T_M (T_M : melting point). However, because of the lack of experimental data at low temperatures with low strain rates, low-temperature region has not been examined well. The map by Frost and Ashby [1] has been drawn based only on the experiment in the power-law breakdown region with \( \dot{\varepsilon} > 10^{-7} \text{ s}^{-1} \) [3, 4]. On the other hand, creep at ambient temperature was observed in hexagonal close-packed (h.c.p.) metals, and ambient-temperature creep was added in the deformation mechanism map of Ti [2]. In Al also, Ishikawa et al. [5] reported different creep behaviour from that by Luthy et al. [4] in the low temperature region. This study, therefore, aimed to re-examine the deformation behaviour in the low-temperature creep region of high-purity Al.
2. Experimental procedure
Creep tests were performed at temperatures from 253 K to 523 K on the samples of 99.999% (5N) Al, 99.99% (4N) Al and 99.5% (1050) Al. The average grain sizes were 140 µm for 5NAl, 100 µm for 4NAl and 50 µm for 1050Al. Creep strains were measured using strain gauges with resolution of $10^{-6}$ with the loading direction parallel to the rolling direction. The steady-state creep rate was estimated through fitting of the logarithmic creep equation [6]:

$$\varepsilon = \varepsilon_i + \varepsilon_p \ln(1 + \beta_p t) + \dot{\varepsilon}_s t,$$

where $\varepsilon$ is the total true strain, $\varepsilon_i$ is the instantaneous strain, $\varepsilon_p$ and $\beta_p$ are parameters characterizing the primary creep region, $\dot{\varepsilon}_s$ is the extrapolated steady-state creep rate, and $t$ is the elapsed time. The optical microscope observations were conducted on the samples before and after the creep test to figure out the microstructual development. The observed sample was polished mechanically by colloidal silica before the creep test.

3. Results
The results of the creep tests are shown in Fig.1 in a double logarithmic plot of strain rate and stress. All specimens showed significant creep above and below room temperature. 5NAl shows stress exponents of about 5.0 below 373 K (0.4$T_M$), and 4.0 above 473 K. 4NAl shows a similar behaviour, and 1050Al shows a slightly larger stress exponent than the materials with higher purity.

Figure 2 is the Arrhenius plot of the three materials at $\sigma/E$ written in the figure. This plot shows two regions with different apparent activation energies, whose border is at about 400 K. All samples show apparent activation energies of 70-80 kJ/mol above this temperature, and 20-35 kJ/mol below this temperature.

Figure 3 shows a double logarithmic plot of creep rate vs. normalized stress for 5NAl with several grain sizes at 300 K; all data points locate on a single line. It is clearly concluded that the creep rate in the low-temperature region is independent of grain size. In addition, this creep phenomenon occurs above the 0.2% proof stress. This means that strain hardening took place before creep testing.

Figure 4 shows an optical micrograph of the deformed sample at room temperature to a strain of 1% at a rate of $10^{-6}$s$^{-1}$. Circles in Fig.4 show that secondary slip system acts. In addition, the other circles show that slip lines propagate into the adjacent grain.

Figure 1. Steady state creep rate for various applied stress normalized by young’s modulus of 5NAl.

Figure 2. The Arrhenius plot for Aluminums with different purity.
4. Discussion

In Fig. 5, the present data are compared to previous creep test data of high purity Al. Luthy et al. [4] and Ishikawa et al. [5] investigated the creep behaviour in the low temperature region. Servi and Grant [7] and Weertman [8] researched the creep behaviour in the high temperature region. As shown in Fig. 4, above 523 K, the data of Weertman and Servi show an activation energy of about 140 kJ/mol which corresponds to the volume diffusion of Al. Present high-temperature data with 70-80 kJ/mol are connecting to their data smoothly. Below 400 K, the plot branches into two lines. One line is reported by Luthy with 80 kJ/mol, which is close to the pipe-diffusion activation energy of Al [9]. On the other hand, Ishikawa reported 20 kJ/mol below 400 K which agrees to the present study well.

Figure 6 plots some selected data of stress and strain rate by Luthy, Ishikawa and us. It is clear that Luthy estimated the activation energy by the data in the power-law breakdown region. Since the effect of temperature dependency of shear modulus is significant in the power-law breakdown region [1], Luthy might have estimated too high activation energies. On the other hand, Ishikawa and we evaluated the creep behaviour near and in the power law region, respectively. Therefore, the activation energy of low-temperature creep of pure Al is concluded to be 20 kJ/mol.

Figure 3. Grain size dependency of the creep behaviour for 5NAl sample crept at 300 K.

Figure 4. Optical micrograph of 5NAl sample after extension of 1% at 300 K under the strain rate of $10^{-9}$ s$^{-1}$.

Figure 5. Arrhenius plot for the present data and creep data obtained from literature.

Figure 6. Double logarithmic plot of selected data from Luthy, Ishikawa, and us below 400 K.
The present study revealed that pure aluminium show creep with stress exponents of 4-5 and activation energies of 20-35 kJ/mol below 400 K. This implies that dislocations contribute to the creep deformation and diffusion hardly contributes. The ambient temperature creep of h.c.p. metals is one of such creep [10]; creep occurs at low temperatures with activation energy of about 20 kJ/mol. Therefore, now we discuss on the differences between the low temperature Al creep and the ambient temperature creep of h.c.p. metals.

First, considering intragranular deformation, h.c.p. metals show only the primary slip system activating and straightly aligned dislocation lines without any tangles. On the other hand, secondary slip line was observed in Al, and its creep proceeds under tangled dislocation structure. In Al, accommodations such as jog formation or cross slip are necessary inside of grains. Activation energies of cross slip and jog formation is about 100 kJ/mol [11] and 14 kJ/mol [12], respectively, and the former may acts as intragrain accommodation.

Next, considering grain boundary deformation, the von Mises law requires at least five slip systems to continue deformation for polycrystalline materials. Since h.c.p. metals have only two slip systems, accommodation has to occur at grain boundaries. However, since Al has 12 slip systems, strain is continuous at grain boundary and accommodation needs not.

5. Conclusion
The low-temperature creep behaviour of pure Al was investigated to re-examine the deformation mechanism map. The extremely low activation energy region was obtained, which has not been reported in the Ashby’s map. The low temperature creep region has the features as follows;
1. The stress exponent is about 4-5.
2. The creep behaviour is independent of grain size.
3. The creep occurs above the 0.2% proof stress.
4. Secondary slip system acts.
5. Slip lines propagate to the adjacent grain.

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