**Title:** Superplasticity at High Strain Rates in Aluminum Alloys

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**Abstract:** The detailed experimental data of microstructural developments during the optimum thermomechanical treatments and superplastic properties in the wide deformation range have been investigated on fourteen P/M aluminum alloys with various chemical matrix compositions and different types of precipitate particles. It was revealed that the differences in both size and volume among the second phase particles, which increase with content of Zr, Cr, Mn or Y, affect on the recrystallization behavior, grain size and superplastic properties of these aluminum alloys. Some of them exhibited high-strain-rate superplasticity, so the optimizing processing methods in this proposal were very powerful to provide the desired structures required for high-strain-rate superplasticity in these aluminum alloys. A theoretical interpretation, based on the experimental data by tensile test and the observed grain sizes, has been revealed that values in activation energy for all alloys are between 140 and 155 kJ/mol, which are similar to that for lattice self-diffusion of aluminum. Each mechanical data of each alloy can be presented by a single equation. It was postulated that superplastic flow in these P/M aluminum alloys was fundamentally controlled by a grain boundary sliding mechanism accommodated by dislocation climb controlled by lattice self-diffusion. However, for the statically recrystallized alloys consisted of the high-angle grain boundaries, the Dom type equation presents n=2, p=2 and D=D, whereas for the dynamically recrystallized alloys with the low-angle grain boundaries, n=3, p=2 and D=D. The consideration for deformation mechanisms with the accommodation helper by such a liquid has been applied on the aluminum alloys, but it remained unclear. The contribution by an accommodation helper to high-strain-rate superplasticity will be the further research subjects, including the investigation of cavitation and fracture.
SUPERPLASTICITY AT HIGH STRAIN RATES 
IN ALUMINUM ALLOYS

FINAL PROGRESS REPORT

KENJI HIGASHI

10 OCTOBER 1996

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OSAKA PREFECTURE UNIVERSITY

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DESIGNATED BY OTHER DOCUMENTATION."
1. FOREWORD

This research program (Grant No. DAAH04-94-G-0070, P00001&00002) designed to investigate superplasticity at high strain rates in aluminum alloys. This program was initiated in order to investigate the understanding of the microstructural development to fine grained superplastic structures by thermomechanical processing and the role of microstructural evolution on the subsequent mechanical properties in fourteen P/M Al-Mg and Al-Cu system alloys including Cr, Mn, Y and Zr.

The investigating experiments consisted of three parts;
1. studying the determination of main alloy compositions of P/M aluminum alloys can exhibit the superplasticity at high strain rates,
2. studying the possibility of grain refinement by an optimum rout of thermomechanical treatment in each P/M aluminum alloy, including the observation for the microstructural developments during the optimum thermomechanical treatment,
3. studying the microstructural evolution during superplastic flow at high strain rates and its relating changes of the mechanical properties, including a determination of the flow stress and the strain rate sensitivity exponent.

Based on the detailed experimental measurements of the mechanical properties by tensile test over a wide deformation range and the microstructural information, the clarification of mechanisms of high-strain-rate superplasticity in P/M aluminum alloys has been tried. Also a theoretical interpretation of the data has been expanded by the new concept of the developing model for deformation mechanisms with the accommodation helper by a liquid phase.

The final objective performed in the present final progress report is to summarize many investigated experiments, to provide a good understanding of the changes in mechanical properties, and to develop the deformation mechanism for high-strain-rate superplasticity.
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      (5) Theoretical Interpretation to Superplastic Deformation Mechanisms
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5. REPORT OF INVENTIONS:   NONE
6. BIBLIOGRAPHY:    NONE
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Table 1: The final chemical compositions of P/M aluminum alloys (wt%).

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Fig. 3: Diffusion-compensated strain rate versus modulus-compensated stress for all P/M aluminum alloys.
4. RESULTS

A. STATEMENT OF THE RESEARCH

The outlines of the investigations in the present research are the following:

(1) Determination of Chemical Compositions of P/M Alloys

(TPR No.1; 1 May 94 ~ 10 July 94)

The chemical compositions of P/M aluminum alloys were nominated in the present work are given as Al-5wt%Cu-Xwt%Zr [0.1<X<1], Al-5wt%Mg-Ywt%Cr [1<Y<2], Al-5wt%Mg-Zwt%Mn [1<Z<3], Al-5wt%Mg-Wwt%Y [0.1<W<2] and Al-5wt%Mg-Vwt%Zr [0.1<V<1]. The characteristics of the precipitates were changed easily by changing the contents of Zr, Cr, Y and Mn. Finally the chemical compositions of two systems P/M aluminum alloys (Al-4.5wt% Cu and Al-5wt%Mg alloys), as shown in Table 1, were selected for using in the present investigation.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Cu</th>
<th>Mg</th>
<th>Zr</th>
<th>Cr</th>
<th>Mn</th>
<th>Y</th>
<th>Fe</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-4.5Cu-0.2Zr</td>
<td>4.33</td>
<td>4.29</td>
<td>0.18</td>
<td>0.81</td>
<td>0.02</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
</tr>
<tr>
<td>Al-4.5Cu-0.3Zr</td>
<td>4.29</td>
<td>4.29</td>
<td>0.32</td>
<td>0.07</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
<tr>
<td>Al-4.5Cu-0.8Zr</td>
<td>4.29</td>
<td>4.29</td>
<td>0.81</td>
<td>0.07</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
<tr>
<td>Al-5Mg-0.2Zr</td>
<td>4.80</td>
<td>4.80</td>
<td>0.22</td>
<td>0.07</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
<tr>
<td>Al-5Mg-0.5Zr</td>
<td>4.82</td>
<td>4.82</td>
<td>0.48</td>
<td>&lt;0.01</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
<tr>
<td>Al-5Mg-0.8Zr</td>
<td>4.76</td>
<td>4.76</td>
<td>0.84</td>
<td>&lt;0.01</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
<tr>
<td>Al-5Mg-0.4Cr</td>
<td>4.78</td>
<td>4.78</td>
<td>0.36</td>
<td>&lt;0.01</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
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<tr>
<td>Al-5Mg-1.0Cr</td>
<td>4.74</td>
<td>4.74</td>
<td>0.97</td>
<td>&lt;0.01</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
<tr>
<td>Al-5Mg-1.5Cr</td>
<td>4.92</td>
<td>4.92</td>
<td>1.39</td>
<td>&lt;0.01</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
<tr>
<td>Al-5Mg-1.5Mn</td>
<td>4.80</td>
<td>4.80</td>
<td>1.48</td>
<td>&lt;0.01</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
<tr>
<td>Al-5Mg-2.2Mn</td>
<td>4.80</td>
<td>4.80</td>
<td>2.24</td>
<td>&lt;0.01</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
<tr>
<td>Al-5Mg-0.3Y</td>
<td>6.26</td>
<td>6.26</td>
<td>0.25</td>
<td>&lt;0.01</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
<tr>
<td>Al-5Mg-0.6Y</td>
<td>5.30</td>
<td>5.30</td>
<td>0.62</td>
<td>&lt;0.01</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
<tr>
<td>Al-5Mg-1.2Y</td>
<td>5.16</td>
<td>5.16</td>
<td>1.22</td>
<td>&lt;0.01</td>
<td>0.03</td>
<td>0.07</td>
<td>0.030</td>
<td>0.070</td>
</tr>
</tbody>
</table>

(2) Observation of Recrystallization Behavior for As-extruded Materials

(TPR No.2; 1 Aug. 94 ~ 10 Oct. 94)

All powders were produced in an industry scale by Technical Research Laboratories of Sumitomo Light Metal Industries Ltd. in Japan. The atomized powders were sieved and the powders below 200 μm were used for extrusion.
Subsequently, the powders were compacted in an aluminum capsule with a diameter of 63.5 mm and degassed in a vacuum chamber (about $10^{-3}$ Torr) at 773 K for $3.6 \times 10^3$ ks. The compact billets were extruded into the bar of 6 mm thickness and 30 mm width with a reduction ratio of 26:1. The operating temperatures for Al-4.5wt% Cu and Al-5wt%Mg systems alloys were 653 and 703 K respectively.

The powder metallurgically processed aluminum alloys consist of two groups, statically recrystallized and dynamically recrystallized alloys. It is generally reported that dynamically recrystallized aluminum alloys exhibit superplasticity at higher strain rates than statically recrystallized aluminum alloys.

(3) **Microstructural Evolution and Quantitative Metallographic Measurements**
(TPR No.3; 10 Oct. 94 ~ 31 May. 95)

The detailed quantitative metallographic measurements of the size and the number of the second phase particles were carried out from the microstructures of as-extruded P/M Al-5wt%Mg systems alloys with various contents of Zr, Cr, Mn or Y. But, in cases for all materials, microstructural changes during straining at an optimum superplastic deformation condition could not be observed by the limited time, with the measurement of misorientation angles among grains and the dynamic behavior of the precipitates. I now strongly desire to continue this investigation of the microstructural changes in the near future by some funds from ARO.

(4) **Experimental Measurements of Superplastic Properties and Grain Sizes**
(TPR No.4; 1 June 95 ~ 31 July. 95: TPR No.5; 1 Aug. 95 ~ 30 Sep. 95;
TPR No.6; 1 Oct. 95 ~ 30 Nov. 95: TPR No.7; 1 Dec. 95 ~ 31 Jan. 96)

A determination of the flow characteristics and the elongation to failure in terms of the variation of strain rate with stress, temperature etc. was curried out
for all fourteen P/M aluminum alloys in a wide deformation conditions. Also the variation of grain size with testing temperature was investigated for all materials.

(5) Theoretical Interpretation to Superplastic Deformation Mechanisms

(TPR No.8; 1 Feb. 96 ~ 31 July. 96)

A theoretical interpretation, based on the experimental mechanical data by tensile test and the observed grain sizes, was considered in the form of the Dorn type constitute equation. Furthermore in order to test the new model of the accommodation process experimentally, the tensile tests and the EDS investigation and in-situ TEM observations at high temperatures near to the incipient melting point were performed for P/M aluminum alloys.

B. SUMMARY

This research leads to many important conclusions, are the following:

- All microstructures of as-extruded Al-Mg-Cr, Al-Mg-Mn and Al-Mg-Y systems alloys had already recrystallized statically, whereas those of Al-Cu-Zr and Al-Mg-Zr systems alloys with higher content of Zr remained unrecrystallized subgrain structures. The microstructures of Al-Cu-Zr and Al-Mg-Zr systems alloys with lower content of Zr consists of the mixing structures of large unrecrystallized subgrains and fine fully recrystallized grains.
- The sizes of the second phase particles (precipitates and inclusions) in Al-Mg alloys including Mn, Cr or Y are larger than those in another alloy with Zr. Also the size, the number and the volume of the second phase particles increase with content of Zr, Cr, Mn or Y. The distribution in the second phase particles for both Al-Mg and Al-Cu alloys with Zr is more inhomogeneous than that for the other alloys with Mn, Cr and Y.
• The detailed quantitative metallographic measurements from the microstructures of as-extruded P/M Al-5wt%Mg systems alloys with various contents of Zr, Cr, Mn or Y revealed that the size of the particles in all Al-Mg-Zr system alloys was one order magnitude finer than that in other Al-Mg system alloys with Cr, Mn or Y, and that the values in diameter for the maximum numbers of the particles were about 30 nm for the Al-Mg and Al-Cu system alloys with Zr, about 0.2 μm for the Al-Mg-Cr and Al-Mg-Y system alloys, and about 0.375 μm for the Al-Mg-Mn system alloys.

• The achieved minimum values in grain size with increasing of the contents of additional elements of Cr, Mn, Y and Zr to P/M aluminum alloys were about 1 μm for Al-Mg and Al-Cu systems alloys with Zr, about 3 μm for Al-Mg systems including Cr and Mn, and about 8 μm for Al-Mg-Y system alloys, as shown in Fig. 1. So an addition of Zr has a bigger contribution to improve a refinement of grain size in P/M Al-Mg system alloys than that of other elements of Mn, Cr and Y.

![Graph showing grain size vs. content of additional elements](image)

Fig. 1: The changes of the grain sizes in P/M aluminum alloys with the contents of additional elements of Cr, Mn, Y and Zr.
Table 2: Superplastic properties of P/M aluminum alloys.

<table>
<thead>
<tr>
<th>Material</th>
<th>Temp. (K)</th>
<th>Strain Rate (s^{-1})</th>
<th>Stress (MPa)</th>
<th>m Value</th>
<th>Max. El(%)</th>
<th>Grain Size* (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-4.5Cu-0.2Zr</td>
<td>798</td>
<td>10^{-4}</td>
<td>4</td>
<td>0.4</td>
<td>100</td>
<td>15</td>
</tr>
<tr>
<td>Al-4.5Cu-0.3Zr</td>
<td>698</td>
<td>3x10^{-3}</td>
<td>10</td>
<td>0.3</td>
<td>325</td>
<td>2</td>
</tr>
<tr>
<td>Al-4.5Cu-0.8Zr</td>
<td>773</td>
<td>10^{-2}</td>
<td>8</td>
<td>0.3</td>
<td>600</td>
<td>1</td>
</tr>
<tr>
<td>Al-5Mg-0.2Zr</td>
<td>848</td>
<td>2x10^{-4}</td>
<td>1</td>
<td>0.5</td>
<td>570</td>
<td>10</td>
</tr>
<tr>
<td>Al-5Mg-0.5Zr</td>
<td>748</td>
<td>10^{-1}</td>
<td>30</td>
<td>0.3</td>
<td>560</td>
<td>1</td>
</tr>
<tr>
<td>Al-5Mg-0.8Zr</td>
<td>773</td>
<td>10^{-1}</td>
<td>21</td>
<td>0.3</td>
<td>570</td>
<td>1</td>
</tr>
<tr>
<td>Al-5Mg-1.0Cr</td>
<td>848</td>
<td>10^{-4}</td>
<td>2</td>
<td>0.5</td>
<td>240</td>
<td>30</td>
</tr>
<tr>
<td>Al-5Mg-1.5Cr</td>
<td>848</td>
<td>10^{-2}</td>
<td>2</td>
<td>0.5</td>
<td>620</td>
<td>3</td>
</tr>
<tr>
<td>Al-5Mg-1.5Mn</td>
<td>823</td>
<td>3x10^{-3}</td>
<td>3</td>
<td>0.5</td>
<td>390</td>
<td>5</td>
</tr>
<tr>
<td>Al-5Mg-2.2Mn</td>
<td>823</td>
<td>6x10^{-3}</td>
<td>4</td>
<td>0.5</td>
<td>570</td>
<td>3</td>
</tr>
<tr>
<td>Al-5Mg-0.3Y</td>
<td>848</td>
<td>10^{-4}</td>
<td>0.5</td>
<td>0.5</td>
<td>570</td>
<td>10</td>
</tr>
<tr>
<td>Al-5Mg-0.6Y</td>
<td>823</td>
<td>4x10^{-4}</td>
<td>2</td>
<td>0.5</td>
<td>435</td>
<td>9</td>
</tr>
<tr>
<td>Al-5Mg-1.2Y</td>
<td>848</td>
<td>10^{-3}</td>
<td>1</td>
<td>0.5</td>
<td>775</td>
<td>8</td>
</tr>
</tbody>
</table>

* The values in grain size are obtained after annealing at superplastic temperatures for 30 min.

- Tensile tests revealed that all alloys exhibited superplasticity in a strain rate range from 10^{-4} to 10^{-1} s^{-1}, as shown in Table 2 and some materials, which had the grain sizes of less than 3 μm, exhibited high-strain-rate superplasticity between nearly 10^{-2} and 10^{-1} s^{-1}.

- Three Al-Mg-Cr, Al-Mg-Mn and Al-Mg-Y systems alloys, also Al-Cu-Zr and Al-Mg-Zr systems alloys with lower content of Zr with a statically recrystallized structure, were the statically recrystallized superplastic materials, whereas Al-Cu-Zr and Al-Mg-Zr systems alloys with higher content of Zr remained unrecrystallized subgrain structures were the dynamically recrystallized superplastic materials.

- An improvement in optimum superplastic strain rate for P/M aluminum alloys is resulting from the refinement of grains with increasing of Cr, Mn, Zr and Y contents. The result obtained in the present work seems to be consistent with
that reported in other aluminum alloys produced by the different processing routes, as shown in Fig. 2.

![Graph showing superplastic strain rate vs. inverse grain size for aluminum alloys](image)

**Fig. 2:** Grain size dependence of superplastic strain rate in P/M aluminum alloys.

- The actively controlled processing methods in both size and volume among the second phase particles affected on the recrystallization behavior, the refinement of grain size and were very powerful to provide the desired structures required for high-strain-rate superplasticity in these P/M aluminum alloys.
- A theoretical interpretation revealed that values in activation energy for all alloys were between 140 and 155 kJmol\(^{-1}\), which were similar to that for lattice self-diffusion of aluminum. Superplastic flow in these P/M aluminum alloys was fundamentally controlled by a grain boundary sliding mechanism accommodated by dislocation climb controlled by lattice self-diffusion.
- Each mechanical data of each statically or dynamically recrystallized superplastic alloy could be presented by a single equation, as shown in Fig. 3. The Dorn typed equation, given by the following:

$$\dot{\varepsilon} = \frac{AGb(b)}{kT} \left( \frac{\sigma - \sigma_0}{G} \right)^n D_0 \exp \left( \frac{-Q}{RT} \right)$$

presented $n=2$, $p=2$ and $D=D_L$ (where $D=D_0 \exp(-Q/RT)$) for the statically recrystallized superplastic alloys consisted of the high-angle grain boundaries, whereas, $n=3$, $p=2$ and $D=D_L$ for the dynamically recrystallized superplastic alloys with the low-angle grain boundaries.

![Graph](image)

Fig. 3: Diffusion-compensated strain rate versus modulus-compensated stress for all P/M aluminum alloys.
• The consideration for deformation mechanisms with the accommodation helper by such a liquid has been applied on the data of the aluminum alloys, and the superplastic elongation strongly depended on the refinement of grain structures and the accommodation process by the presence of the helper, such as a liquid phase. But the contribution by an accommodation helper to high-strain-rate superplasticity remained unclear. A summary of the optimum superplastic temperatures, the solidus temperatures and the incipient melting temperatures of a number of P/M aluminum alloys is shown in Table 3.

<table>
<thead>
<tr>
<th>Material</th>
<th>Superplastic Temp.(K)</th>
<th>Solidus Temp.(K)</th>
<th>Incipient Melting Temp.(K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-4.5Cu-0.2Zr</td>
<td>798</td>
<td>845</td>
<td>818</td>
</tr>
<tr>
<td>Al-4.5Cu-0.3Zr</td>
<td>698</td>
<td>847</td>
<td>818</td>
</tr>
<tr>
<td>Al-4.5Cu-0.8Zr</td>
<td>773</td>
<td>847</td>
<td>818</td>
</tr>
<tr>
<td>Al-5Mg-0.2Zr</td>
<td>848</td>
<td>852</td>
<td></td>
</tr>
<tr>
<td>Al-5Mg-0.5Zr</td>
<td>748</td>
<td>852</td>
<td></td>
</tr>
<tr>
<td>Al-5Mg-0.8Zr</td>
<td>773</td>
<td>852</td>
<td></td>
</tr>
<tr>
<td>Al-5Mg-0.4Cr</td>
<td>848</td>
<td>854</td>
<td></td>
</tr>
<tr>
<td>Al-5Mg-1.0Cr</td>
<td>848</td>
<td>856</td>
<td></td>
</tr>
<tr>
<td>Al-5Mg-1.5Cr</td>
<td>848</td>
<td>855</td>
<td></td>
</tr>
<tr>
<td>Al-5Mg-1.5Mn</td>
<td>823</td>
<td>847</td>
<td></td>
</tr>
<tr>
<td>Al-5Mg-2.2Mn</td>
<td>823</td>
<td>843</td>
<td></td>
</tr>
<tr>
<td>Al-5Mg-0.3Y</td>
<td>848</td>
<td>845</td>
<td></td>
</tr>
<tr>
<td>Al-5Mg-0.6Y</td>
<td>823</td>
<td>843</td>
<td></td>
</tr>
<tr>
<td>Al-5Mg-1.2Y</td>
<td>848</td>
<td>842</td>
<td></td>
</tr>
</tbody>
</table>

• The simple and optimum thermo-mechanical treatment routes were established for P/M aluminum alloys, which will lead the cost effective production method for other materials could exhibit high-strain-rate superplasticity.

• A good understanding of the changes in mechanical properties during superplastic flow at high strain rates was provided, which could permit to develop the mechanism for high strain rate superplasticity based on microstructural view.
• It is still uncertain that the presence of the accommodation helper is unique requirement to achieve high-strain-rate superplasticity in P/M aluminum alloys, then it will be the further research subjects, including the investigation of cavitation and fracture.

C. LIST OF THE PUBLICATIONS

1. Effect of Temperature on the Mechanical Properties of Mechanically-Alloyed Materials at High Strain Rates
   K.Higashi, T.G.Nieh and J.Wadsworth

2. Mechanisms of High Strain-Rate Superplasticity of Al-14mass%Ni-14mass%Mm (Misch Metal) Alloy Produced from Amorphous Powders
   K.Higashi, T.Mukai, A.Uoya, A.Inoue and T.Masumoto

3. Interface Structure of Si3N4-Whisker-Reinforced Al-Mg-Si (Al alloy 6061) Composites Studied by High-resolution Electron Microscopy
   H-G. Jeong, K. Hiraga, M. Mabuchi and K. Higashi

4. A Quantitative Analysis of Cavitation in Al-Cu-Mg Metal Matrix Composites Exhibiting High Strain Rate Superplasticity
   S.Wada, M.Mabuchi, K.Higashi and T.G.Langdon
5. Introduction to High Strain Rate Superplasticity
   K. Higashi

6. High-strain-rate Superplasticity in Magnesium Matrix Composites Containing Mg_{2}Si Particles
   M. Mabuchi and K. Higashi

7. Strengthening Mechanisms of Mg-Si Alloys
   M. Mabuchi and K. Higashi

8. Superplasticity at a Low Temperature below 0.5Tm in an AZ91 Magnesium Alloy Processed by ECAE
   Mabuchi, H. Iwasaki, K. Yanase, T. Mori and K. Higashi

9. High-Strain-Rate Superplasticity in Metallic Materials and Its Innovation in Ceramic Materials (Review)
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5. REPORT OF INVENTIONS: NONE

6. BIBLIOGRAPHY: NONE