QUARTERLY REPORT FOR THE PERIOD 9/1/94 TO 12/30/94

NANOSTRUCTURED BEARING ALLOY STUDIES
[ONR N00014-94-1-0579]

SUBMITTED TO:

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PROCESSING (consolidation)

Introduction

Earlier results from this program have shown that the commercial practice that were
developed to handle reactive powders such as titanium, is not adequate for nano powders. Using
such practice, oxygen content up to 12% was observed in consolidated nano steel samples. A series
of improvements has been made recently in powder handling and consolidation. A more inert
atmosphere in the glove box is achieved by incorporation of a diffusion pump and an argon purifying
line. The powder is heat treated in a 4% hydrogen-argon mixture in the vacuum press prior to
compaction. These changes and resulting reduction in oxygen content in the powder compact were
presented in the Second Quarterly Report (October 1994). An additional improvement was made
during this reporting period which involved the construction and use of a hydrogen retort to further
reduce the oxygen and carbon content in the nano powder prior to consolidation. Several
consolidations were also made to evaluate the effectiveness of this improvement.

In addition to monitoring the oxygen content and hydrogen content in these new powder
compacts, hardness measurements were also made to determine the hardness response of the nano
steels. Consolidated materials have also been given to Professor John Morral for physical metallurgy
and heat treatment studies.

Results

The carbon, oxygen and hydrogen contents in the powder compacts are given in Table 1. Of
interest is a comparison of the chemical data in the compacts which had undergone pre-compaction
treatment in 4% hydrogen (Compacts 3E and 4F) with those treated in the hydrogen retort
(Compacts 4G and 5H). It can be seen from Table 1 that hydrogen treatment for 2 hours at 400°C
in the retort was ineffective in further reducing the carbon and oxygen content but significant
increased the hydrogen content relative to the compact which had undergone pre-compaction
treatment in 4% hydrogen only. Hydrogen heat treatment of nano M50 powder in the retort is now
in progress to define the optimum conditions for reducing both the carbon and oxygen contents.

The hardness levels of selected powder compacts are given in Table 2. The data show that
very high hardness levels can be achieved both in nano M50 steel and the iron silicon alloy. The
origin of the high hardness is not known at present and will be subject for investigation in this
program. A comparison of the hardness level in Compact 4F and 4G shows that reducing the
compaction temperature from 850°C to 700°C increased the hardness of iron silicon alloy by a
factor of two. This observation suggests that the high hardness may be associated with the fine scale
microstructure produced by consolidation at the lower temperature.
<table>
<thead>
<tr>
<th>Powder Batc</th>
<th>Cover</th>
<th>Gloe Bo</th>
<th>Pre-Co Treat ent</th>
<th>Car on</th>
<th>O en</th>
<th>Hydro en</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 (M50)</td>
<td>1A, 1B</td>
<td>Mech. Pump Ar Atms.</td>
<td>None</td>
<td>5.6</td>
<td>11.9</td>
<td>-</td>
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<tr>
<td>2 (M50)</td>
<td>2C, 2D</td>
<td>Mech. Pump Ar Atms.</td>
<td>None</td>
<td>8.2</td>
<td>12.3</td>
<td>-</td>
</tr>
<tr>
<td>3 (M50)</td>
<td>3E</td>
<td>Diffusion Pump Purified Air</td>
<td>850F 4h in 4% H₂ Atms. in Vac. Press</td>
<td>6.2</td>
<td>3.1</td>
<td>17</td>
</tr>
<tr>
<td>4a (Fe-1.5 Si)</td>
<td>4F</td>
<td>Diffusion Pump Purified Air</td>
<td>428C 27h in 4% H₂ Atms. in Vac. Press</td>
<td>3.3</td>
<td>1.2</td>
<td>-</td>
</tr>
<tr>
<td>4b (Fe-1.5 Si)</td>
<td>4G</td>
<td>Mech. Pump Purified Air</td>
<td>400C 2h in H₂ Retort, Heating up in 4% H₂ Atms. in Vac. Press</td>
<td>2.5</td>
<td>3.4</td>
<td>-</td>
</tr>
<tr>
<td>5 (M50)</td>
<td>5H</td>
<td>Mech. Pump Purified Air</td>
<td>400C 2h in H₂ Retort, Heating up in 4% H₂ Atms. in Vac. Press</td>
<td>6.2</td>
<td>5.8</td>
<td>1700</td>
</tr>
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</table>
Table 2
Hardness of Nano Steel Compacts

<table>
<thead>
<tr>
<th>Compact Number</th>
<th>Alloy</th>
<th>Consolidation Conditions</th>
<th>Hardness (Rockwell C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3E</td>
<td>M50</td>
<td>850°C/275MPa</td>
<td>70</td>
</tr>
<tr>
<td>4F</td>
<td>Fe-1.5Si</td>
<td>850°C/275MPa</td>
<td>30</td>
</tr>
<tr>
<td>4G</td>
<td>Fe-1.5Si</td>
<td>700°C/275MPa</td>
<td>60</td>
</tr>
<tr>
<td>5H</td>
<td>M50</td>
<td>700°C/275MPa</td>
<td>*</td>
</tr>
</tbody>
</table>

* Load ram fractured during consolidation, compact partially dense

PHYSICAL METALLURGY OF NANOSTRUCTURED M50 STEEL

Literature Survey

An initial survey of the literature on the physical metallurgy of M50 steel and the effect of grain size on the heat treatment and hardening of steels was completed and is currently being summarized in a report. Little published work was found on these topics. The survey identified only four papers on the physical topics. The survey identified only four papers on the physical metallurgy of M50 steel, two papers on the effect of austenitic grain size on hardenability and four papers related to the grain growth of nanostructured ferrous alloys. Therefore, baseline studies will be required to characterize conventional M50 steels.

Baseline Studies

Studies on conventional M50 steel were begun in order to develop sample preparation and characterization techniques tailored to M50 alloys and to collect baseline data. Characterization techniques being developed are dilatometry, metallography, x-ray diffraction and electron microscopy. In addition to a conventional dilatometer at Pratt & Whitney, a dilatometer with rapid heating and cooling capabilities was located at a private consulting laboratory. We are awaiting details on the cost and availability of this equipment.

Future Work

Information on austenitic grain growth and the cooling transformation kinetics of conventional M50 steel is expected from the baseline studies. These results will be compared with those obtained from nanostructured materials once they are available. The goal is to establish the effect of nanocrystalline grain size on the hardenability, martensite microstructure, carbide distribution, retained austenite, and hardness of M50 steel.