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**Declarations under Rule 4.17**:

— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*

*[Continued on next page]*

(54) **Title**: NICKEL BASED ALLOY USEFUL FOR VALVE SEAT INSERTS

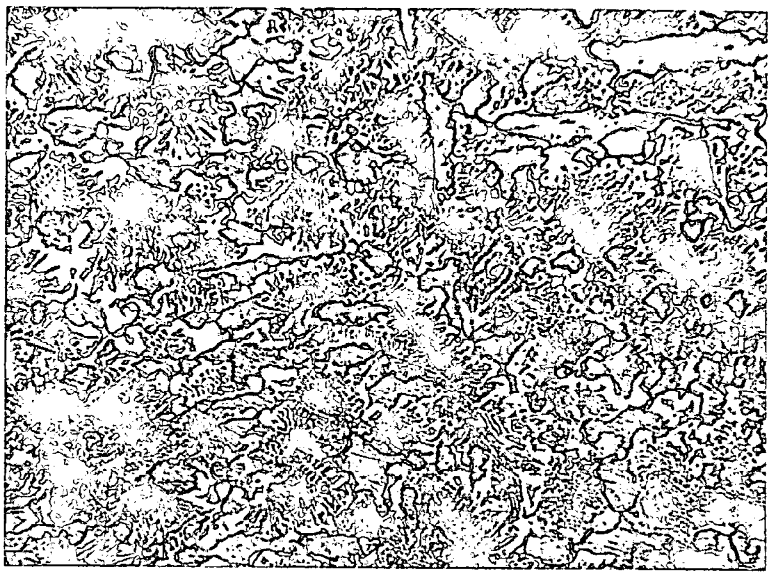

FIG. 1

(57) **Abstract**: Disclosed herein is a nickel based alloy comprising, in weight percentage: carbon from about 0.5 to about 1.5; chromium from about 25 to about 35; tungsten from about 12 to about 18; iron from about 3.5 to about 8.5; molybdenum from about 1 to about 8; manganese up to about 0.50; silicon up to about 1.0; and the balance nickel and incidental impurities. The alloy is suitable for valve seat insert applications in internal combustion engines.

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— *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))*

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# NICKEL BASED ALLOY USEFUL FOR VALVE SEAT INSERTS

## FIELD OF THE INVENTION

**[0001]** The invention relates to nickel based alloys having high hardness and compressive yield strength. Such alloys are especially useful for engine parts such as valve seat inserts.

## DESCRIPTION OF THE RELATED ART

**[0002]** Nickel based valve seat insert alloys generally have wear resistance, heat resistance, and corrosion resistance superior to those of high alloy steels, and are therefore often used as materials for structural members serving under severe conditions, such as valve seat inserts. Known nickel based alloys used for exhaust valve seat inserts, such as an alloy identified as J96 and marketed by L. E. Jones Company, have relatively good characteristics, including good hardness and compressive yield strengths. Another alloy marketed by L. E. Jones is J89, details of which are provided in U.S. Patent No. 6,482,275, the disclosure of which is hereby incorporated by reference. In general, the J89 alloy includes, in weight percent (as used herein "percent" and "%" refer to percent by weight unless otherwise indicated) , 2.25 to 2.6 % C, up to 0.5 % Mn, up to 0.6 % Si, 34.5 to 36.5 % Cr, 4.00 to 4.95 % Mo, 14.5 to 15.5 % W, 5.25 to 6.25 % Fe, balance Ni plus incidental impurities.

## SUMMARY

**[0003]** Disclosed herein is a nickel based alloy comprising, in weight percentage: carbon from about 0.5 to about 1.5; chromium from about 25 to about 35; tungsten from about 12 to about 18; iron from about 3.5 to about 8.5; molybdenum from about 1 to about 8; manganese up to about 0.50; silicon up to about 1.0; and the balance nickel and incidental impurities. The alloy is suitable for valve seat insert applications in internal combustion engines.

BRIEF DESCRIPTION OF THE DRAWING FIGURES

**[0004]** FIG. 1 is an OLM photomicrograph at 500X of J91 in an as-cast condition.

**[0005]** FIG. 2 is an SEM photomicrograph at 500X of J91 in an as-cast condition.

**[0006]** FIG. 3 is a graph of wear at elevated temperature for J3, J130, J160 and J91 alloys.

DETAILED DESCRIPTION

**[0007]** The nickel based alloy described herein (referred to as "the J91 alloy") has been designated to promote machinability while maintaining desired hardness and wear resistance at elevated temperatures. Through adjustments in carbon, chromium, nickel and tungsten contents, it is possible to provide a matrix material which is free of coarse primary carbides yet exhibits desired wear resistance properties. The microstructure of the J91 alloy can be characterized as spheroidal or egg-shaped eutectic domains interspersed with a Ni-rich FCC phase and thus provide desired wear resistance properties without reliance on coarse primary carbides.

**[0008]** In addition to improved machinability and desired hardness, the J91 alloy can exhibit high compressive yield strength, good corrosion resistance and good oxidation resistance.

**[0009]** Before embodiments are explained in detail, it is to be understood that the J91 alloy is not limited in its application to the details of the composition and concentrations of components set forth in the following description. The J91 alloy is capable of other embodiments and of being practiced or being carried out in various ways. Also, it is understood that the phraseology and terminology used herein is for the purpose of description and should not be regarded as limiting. When used herein, the term "about" is intended to include values up to 10 % higher and up to 10 % lower than the numerical value or ranges recited.

**[0010]** While the J91 alloy is designed particularly for use in internal combustion engine valve seat inserts, other applications are feasible. Compared to the J89 alloy, the J91 alloy is based on experimental findings that hardness and compressive yield strength of the nickel based alloys can be obtained by removal of coarse primary carbides and creating an evenly distributed face centered cubic (FCC) nickel-solid solution phase in eutectic reaction phases matrix in which additional strengthening solutes can be introduced.

[0011] Carbon (C) is present in the J91 alloy in an amount ranging from about 0.5 to about 1.5 weight percent of the total alloy; preferably, about 0.95 to about 1.3 weight percent. The J91 alloy surprisingly exhibits wear resistance properties equivalent to that of the J89 alloy but with a much lower carbon content. Whereas the J89 alloy relies on the presence of coarse primary carbides to achieve wear resistance, the J91 alloy which is preferably free of coarse primary carbides can achieve desired wear resistance in an as-cast condition through an improved wear resistant matrix microstructures. By selection of Ni, Cr and W contents it is possible to increase the amount of eutectic structure through ternary eutectic reactions which produce spheroidal or egg-shaped eutectic domains interspersed with a Ni-rich FCC phase.

[0012] Chromium (Cr) is present in the alloy in an amount ranging from about 25 to about 35 weight percent of the total alloy, preferably 27 to 33 weight percent, and more preferably about 28.5 to about 31.5 weight percent of the total alloy. The chromium content can be selected such that the relative amounts of Cr, Ni and W move the J91 alloy closer to the center of the eutectic center point of the Ni-W-Cr ternary phase diagram so as to promote the tendency for intermetallic phase(s) formation between W and Ni. By increasing the amount of uniformly distributed eutectic structures, the matrix material can be made very wear resistant.

[0013] Tungsten (W) is present in the alloy in an amount ranging from about 12 to about 18 weight percent of the total alloy. Preferably, the tungsten content is at least about 14 weight percent and at most about 16 weight percent. A more preferred W content is about 14.5 to about 15.5 %.

[0014] Iron (Fe) is present in the alloy in an amount ranging from about 3.5 to about 8.5 weight percent of the total alloy; preferably, at least about 5 weight percent and at most about 7 weight percent. A preferred Fe content is about 5.25 % to about 8.25%.

[0015] Molybdenum (Mo) is present in the alloy in an amount ranging from about 1 to about 8 weight percent of the total alloy. Generally, greater molybdenum increases alloy hardness and decreases carbide size; however, too much molybdenum may result in a brittle product. The weight percent molybdenum is preferably at least about 2 weight percent and at most about 6.25 weight percent. More preferably, the alloy contains about 4 to 5 weight percent Mo, most preferably the Mo content is about 4.35 % to about 4.95 %.

**[0016]** Manganese (Mn) can be added or present in an amount of up to about 0.5 weight percent of the total alloy. A preferred Mn content is about 0.25 % to about 0.5 %.

**[0017]** Silicon (Si) may be added to or present in the alloy at levels up to about 1.0 weight percent of the total alloy. A preferred Si content is about 0.15 % to about 0.60 %.

**[0018]** The alloy may contain other intentionally added elements up to a total of 1.5 weight percent. Such other elements include cobalt (Co), vanadium (V), titanium (Ti), niobium (Nb), hafnium (Hf), zirconium (Zr), tantalum (Ta), rare earth, yttria (Y), copper (Cu), sulfur (S), phosphorus (P), nitrogen (N) or other elements. For example, the alloy may include up to 0.5% V, up to 0.5% Co, up to 0.03% P, up to 0.03% S.

**[0019]** The balance of the alloy is nickel (Ni) and incidental impurities. Generally, the alloy contains at least about 30 weight percent nickel. A preferred Ni content is about 35 to about 45 %. Thus, the alloy preferably consists essentially of C, Cr, W, Mo, Fe, Ni, Mn and Si. As used herein "consisting essentially of" excludes additions which adversely affect machinability and wear properties of the alloy.

**[0020]** At 800°C., the matrix material between the carbides preferably contains a three-phase eutectic composition of the elements Cr--Ni--W, which provides increased strength. The relative concentration of Cr--Ni--W necessary to form a three-phase eutectic composition may be determined by reference to a Cr--Ni--W ternary component phase diagram. Such phase diagrams are shown, for example, on page 3-48 of the ASM Handbook, Copyright 1992, Volume 3, which is herein incorporated by reference.

**[0021]** In a highly preferred embodiment, the alloy comprises:

[0022]

| Element | Weight Percent Range |
|---|---|
| C | 0.95-1.3 |
| Cr | 28.5-31.5 |
| Mo | 4.35-4.95 |
| W | 14.5-15.5 |
| Fe | 5.25-8.25 |
| Si | 0.15-0.6 |
| Mn | 0.25-0.50 |
| V | up to 0.5 |
| Co | up to 0.5 |
| S | up to 0.03 |
| P | up to 0.03 |
| Ni | balance |
| other elements | up to 1.5 |

[0023] Metal parts can be made from the alloy by casting or forming from a powder and sintering, or the alloy can be used as a coating to hardface parts. Preferably, the alloy is manufactured by casting. Casting is a conventional process in which raw materials are added together and melted to liquid state, and then poured into a cast mold.

[0024] Preferably, the metal parts are valve seat inserts made by casting or powder metallurgy for use in internal combustion engines.

[0025] Although the J91 alloy is nickel-based, the thermal expansion coefficient of the alloy tends to be closer to that of iron than nickel. (The thermal expansion coefficient of cast iron is approximately 11.5 x $10^{-6}$ mm/mm °C. at a temperature of 25-600°C.) This is beneficial because the valve seat insert tends to be much hotter than the surrounding material when the engine is operating. If the thermal expansion coefficient of the valve insert alloy closely matches that of the cylinder head alloy, this enables the insert and cylinder head to expand at the same rate, thereby improving insert retention characteristics.

**[0026]** The J91 alloy has good high temperature compressive yield strength which increases wear resistance and decreases material yielding during operation. Decreased yielding serves to improve insert retention. Preferably, the alloy has a compressive yield strength of at least about 110 thousand pounds per square inch (KSI) at room temperature; more preferably, at least about 130 KSI at room temperature.

**[0027]** Increased hot hardness contributes to improved wear resistance and provides a safety factor for inserts which run beyond the normal operating temperature.

EXAMPLE

**[0028]** Comparative properties of the J91 and J89 alloys are set forth in the following tables and discussion.

Typical Microstructures

**[0029]** The J91 alloy possesses a matrix composed of eutectic reaction phases along with a small amount of randomly distributed FCC nickel solid solution phase. The nickel solid solution phase is distributed along the grain boundaries of eutectic phases. An optical light microscope (OLM) photomicrograph and a scanning electron microscope (SEM) photomicrograph exhibiting typical J91 microstructures are depictured in Figures 1 and 2, respectively. A heat of J89 (Heat No. 7K17K) and a heat of J91 (Heat No. 8L15XA) were employed for the optical light microscopic microstructural characterization. In addition, a heat of J91 (Heat No. 7G10XA) and a heat of J89 (7K17K) were employed for the scanning electron microscopic microstructural characterization. The composition of above three heats involved is summarized in the Table 1.

[0030] Table 1. Composition of J89 and J91 heats applied for the microstructural characterization.

| Alloy/Heat No. | C | Si | Mn | Cr | Mo | W | Fe | Ni |
|---|---|---|---|---|---|---|---|---|
| J89/7K17K | 2.25 | 0.20 | 0.39 | 35.12 | 4.48 | 15.00 | 5.69 | 36.49 |
| J91/8L15XA | 1.19 | 0.20 | 0.52 | 30.51 | 4.44 | 14.92 | 7.19 | 40.72 |
| J91/7G10XA | 1.21 | 0.16 | 0.02 | 30.54 | 4.88 | 14.20 | 4.47 | 41.32 |

Samples Used for Hot Hardness Measurement

[0031] Composition of the heats used for making hot hardness measurement samples for alloys J89 and J91 are summarized in Table 2.

[0032] Table 2. Composition of alloy / heat numbers of J89 and J91 used for the hot hardness tests.

| Alloy/Heat No. | C | Si | Mn | Cr | Mo | W | Fe | Ni |
|---|---|---|---|---|---|---|---|---|
| J89/4E18D | 2.40 | 0.39 | 0.26 | 34.92 | 4.38 | 14.90 | 5.93 | 36.64 |
| J91/8D02Q | 0.98 | 0.46 | 0.22 | 30.55 | 4.36 | 15.25 | 6.95 | 41.06 |

Material Properties

[0033] Typical bulk hardness of alloy J91 is Rockwell C (HRC) of 48 to 52, preferably about 49 to 51. Thus, alloy J91 possesses a bulk hardness in between that for J96 (HRC 40) and J89 (HRC 55).

[0034] A comparison of hot hardness (in Vickers HV10 units) for J89, J91, and J96 (for alloys summarized in Table 2) is summarized in Table 3. J91 was found to possess a significant greater hot hardness compared to J96, even though J91 does not contain coarse primary carbide in its microstructures.

[0035] Table 3. A comparison of hot hardness among alloys J89, J91, and J96.

| Temperature (°F) | J89 (HV10) | J91 (HV10) | J96 (HV10) |
|---|---|---|---|
| 75 | 611 | 456 | 367 |
| 200 | 641 | 426 | 360 |
| 400 | 555 | 387 | 325 |
| 600 | 602 | 375 | 337 |
| 800 | 532 | 372 | 322 |
| 1000 | 556 | 366 | 338 |
| 1200 | 504 | 349 | 292 |
| 1400 | 463 | 318 | 250 |
| 1600 | 320 | 220 | 153 |

Samples Used for Compression Yield Strength Test

[0036] The J89 and J91 samples used for compression tests are set forth in Table 4.

[0037] Table 4

| Alloy | C | Si | Mn | Cr | Mo | Fe | W | Ni |
|---|---|---|---|---|---|---|---|---|
| J89 | 2.51 | 0.56 | 0.48 | 36.47 | 4.15 | 6.7 | 15.44 | 33.69 |
| J91 | 1.33 | 0.24 | 0.1 | 30.29 | 4.81 | 8.69 | 14.15 | 40.39 |

[0038] A comparison of compressive yield strength of J89, J91, and J96 is shown in Table 5. It is clearly shown that J91 possesses compressive yield strengths for the applied temperature range between that for alloys J89 and J96.

**[0039]** Table 5. Comparison of compressive yield strength (KSI) among J89, J91, and J96.

| Temperature (°F) | J89 (KSI) | J91 (KSI) | J96 (KS |
|---|---|---|---|
| 75 | 130.0 | 113.2 | 65. |
| 600 | 115.4 | 88.4 | 61. |
| 800 | 112.2 | 83.7 | 64. |
| 1000 | 115.4 | 93.1 | 66. |

Samples Used for Linear Thermal Expansion Coefficient Measurement

**[0040]** A heat of J89 (4E18D) and a heat of J91 (7G10XA) were used for carrying out the thermal expansion coefficient measurement. Compositions for the two involved heats are summarized in Table 6.

**[0041]** Table 6. Composition of J89 and J91 heats applied for thermal expansion coefficient test.

| Alloy/Heat No. | C | Si | Mn | Cr | Mo | W | Fe | Ni |
|---|---|---|---|---|---|---|---|---|
| J89/4E18D | 2.40 | 0.39 | 0.26 | 34.92 | 4.38 | 14.90 | 5.93 | 36.64 |
| J91/7G10XA | 1.21 | 0.16 | 0.02 | 30.54 | 4.88 | 14.20 | 4.47 | 41.32 |

**[0042]** Results of the thermal coefficient measurement of alloys J89 and J91 with above described heats are summarized in Table 7.

**[0043]** Table 7. Thermal expansion coefficient ($x10^{-6}$ mm/mm °C) for alloys J89 and J91

| Alloy/Heat No. | 25-200°C | 25-300°C | 25-400°C | 25-500° | 25-600°C |
|---|---|---|---|---|---|
| J89/4E18D | 10.32 | 11.07 | 11.55 | 11.95 | 12.38 |
| J91/7G10XA | 10.95 | 11.63 | 12.15 | 12.52 | 13.01 |

**[0044]** Practically, the thermal expansion coefficient of J91 was only slightly greater than that for J89. Such a low thermal expansion coefficient is favorable for heavy duty engine valve seat insert applications.

Wear Resistance

[0045] The wear resistance of alloy J91 under engine wear conditions is expected to be similar to J89. A comparison of wear resistance as a function of test temperature for J91, J3, J130, and J160 vs Pyromet 31 V valve material is exhibited in Figure 3 and Table 8, respectively.

[0046] It is clearly shown that in the exhaust temperature range, J91 exhibited overall the least total materials wear among the four materials pairs evaluated. Within the lower test temperature range (ambient to 250°C), J91 showed a similar wear resistance to alloys J130 and J160 when paired with Pyromet 31 V valve material.

[0047] Table 8. Summary of the Plint wear test results.

| Total Materials Wear of J130, J160, J3 and J91 (pin) vs Pyromet 31 (plate) | | | | |
|---|---|---|---|---|
| Temp (°C) | J3 | J130 | J160 | J91 |
| 20 | 1.0 | 3.9 | 5.3 | 4.7 |
| 200 | 2.9 | 5.1 | 4.7 | 5.5 |
| 250 | 5.0 | 4.9 | 5.5 | 3.0 |
| 300 | 3.6 | 3.4 | 3.3 | 3.7 |
| 350 | 5.7 | 3.3 | 4.2 | 2.9 |
| 400 | 4.6 | 3.1 | 3.7 | 2.1 |
| 450 | 5.2 | 1.9 | 4.3 | 1.6 |
| 500 | 1.9 | 0.8 | 1.6 | 2.2 |

[0048] It should be appreciated that the alloys of the present invention are capable of being incorporated in the form of a variety of embodiments, only a few of which have been described above. The invention may be embodied in other forms without departing from its spirit or essential characteristics. The described embodiments are considered in all respects only as illustrative and not restrictive, and the scope of the invention is, therefore, indicated by the appended claims rather than by the foregoing description. All changes which come within the meaning and range of equivalency of the claims are to be embraced within their scope.

WHAT IS CLAIMED IS:

1. A nickel based alloy comprising, in weight percentage: carbon from about 0.5 to about 1.5; chromium from about 25 to about 35; tungsten from about 12 to about 18; iron from about 3.5 to about 8.5; molybdenum from about 1 to about 8; manganese up to about 0.50; silicon up to about 1.0; nickel from about 30 to about 45 and incidental impurities.

2. The alloy of Claim 1, wherein the alloy comprises about 0.95 to about 1.30 weight percent carbon.

3. The alloy of Claim 1, wherein the alloy comprises about 28.5 to about 30.5 weight percent chromium.

4. The alloy of Claim 1, wherein the alloy comprises at least about 14.0 weight percent tungsten.

5. The alloy of Claim 1, wherein the alloy comprises at least about 5.0 weight percent iron and at most about 7.0 weight percent iron.

6. The alloy of Claim 1, wherein the alloy comprises about 4.5 weight percent molybdenum.

7. The alloy of Claim 1, wherein the alloy comprises at most about 1.3 weight percent carbon.

8. The alloy of Claim 1, wherein the alloy comprises at most about 32.0 weight percent chromium.

9. The alloy of Claim 1, wherein the alloy comprises at most about 16.0 weight percent tungsten.

10. The alloy of Claim 1, wherein the alloy comprises about 40 to 42 weight percent nickel.

11. The alloy of Claim 1, wherein the relative concentration of Cr, Ni and W is such that a three-phase eutectic composition forms at a temperature of about 800°C.

12. The alloy of Claim 1, wherein the alloy consists essentially of, in weight percentage: carbon from about 0.95 to about 1.3; chromium from about 28.5 to about 31.5; tungsten from about 14.5 to about 15.5; iron from about 5.25 to about 8.25; molybdenum from about 4.35 to about 4.95; manganese from about 0.25 to about 0.5; silicon from about 0.15 to about 0.6; vanadium up to about 0.5, cobalt up to about 0.5, sulfur up to about 0.03, phosphorus up to about 0.03, and nickel from about 38 to about 42 and incidental impurities.

13. The alloy of Claim 1, wherein said alloy is a casting.

14. The alloy of Claim 12, wherein said alloy is a casting.

15. The alloy of Claim 1, wherein said alloy is a valve seat insert for an internal combustion engine.

16. The alloy of Claim 1, wherein the alloy has an as-cast microstructure comprising a wear resistant matrix of spheroidal eutectic domains free of coarse primary carbides.

17. A valve seat insert for use in an internal combustion engine, said valve seat insert made of an alloy consisting essentially of in weight percent: carbon from about 0.5 to about 1.5; chromium from about 25 to about 35; tungsten from about 12 to about 18; iron from about 3.5 to about 8.5; molybdenum from about 1 to about 8; manganese up to about 0.5; silicon up to about 1.0; and the balance nickel and incidental impurities.

18. The valve seat insert of Claim 17, wherein the valve seat insert is a casting.

19. The valve seat insert of Claim 17, wherein the alloy consists essentially of carbon from about 0.95 to about 1.3; chromium from about 28.5 to about 31.5; tungsten from about 14.5 to about 15.5; iron from about 5.25 to about 8.25; molybdenum from about 4.35 to about 4.95; manganese from about 0.25 to about 0.5; silicon from about 0.15 to about 0.6; total of vanadium and cobalt not exceeding 0.5, sulfur not exceeding 0.03, phosphorus not exceeding 0.03, and the balance nickel and incidental impurities.

20. The valve seat insert of Claim 17, having a hardness of about 48 to about 52 Rockwell C.

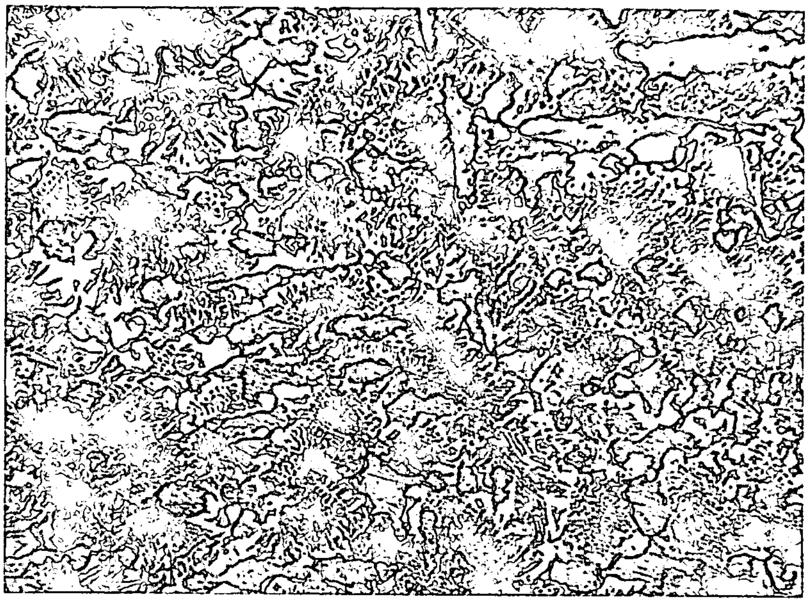

FIG. 1

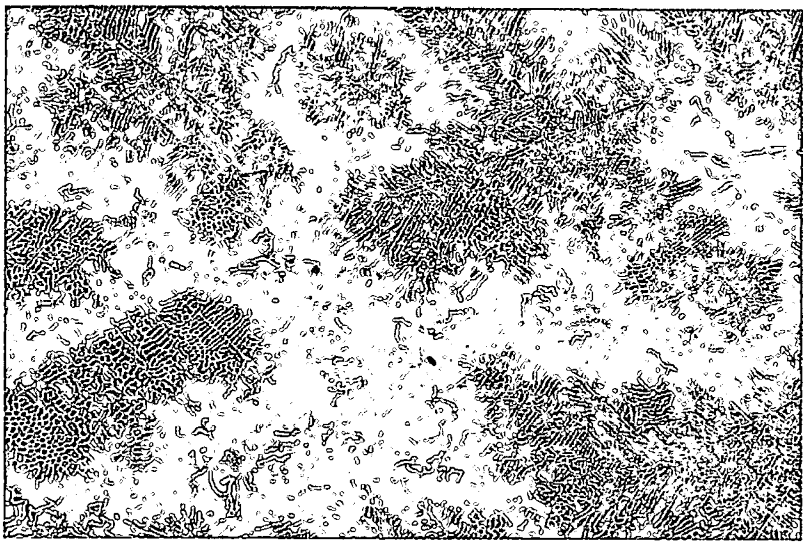

FIG. 2

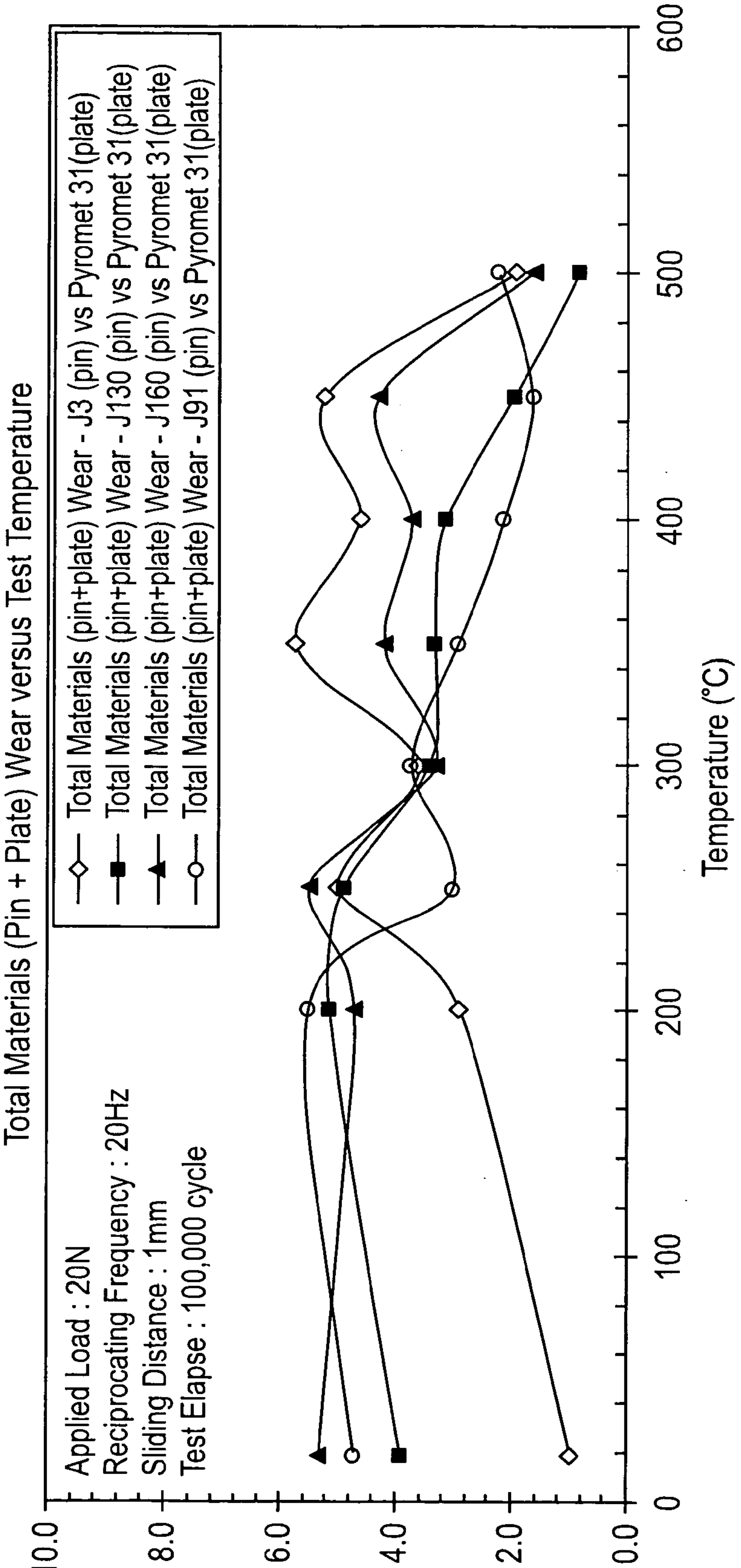
Total Materials (Pin + Plate) Wear versus Test Temperature
Total Materials (pin+plate) Wear - J3 (pin) vs Pyromet 31(plate)
Total Materials (pin+plate) Wear - J130 (pin) vs Pyromet 31(plate)
Total Materials (pin+plate) Wear - J160 (pin) vs Pyromet 31(plate)
Total Materials (pin+plate) Wear - J91 (pin) vs Pyromet 31(plate)
Applied Load : 20N
Reciprocating Frequency : 20Hz
Sliding Distance : 1mm
Test Elapse : 100,000 cycle
Materials Loss (mg)
10.0
8.0
6.0
4.0
2.0
0.0
Temperature (°C)
0
100
200
300
400
500
600

FIG. 3