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FINE-SCALE MECHANICAL PROPERTIES OF SLIDING SOLIDS(U)
LANCASTER UNIV (ENGLAND) DEPT OF PHYSICS H M POLLOCK
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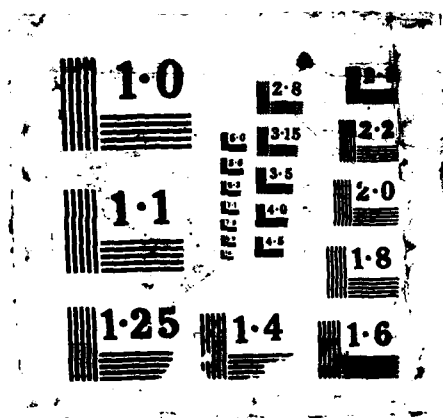
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February 28, 1986

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Title: FINE-SCALE MECHANICAL PROPERTIES OF SLIDING SOLIDS

Principal investigator: Dr. H. M. Pollock, Department of Physics

Contractor: University of Lancaster (atten. Finance Officer),
Lancaster, England LA1 4YW

Contract number: DAJA 45-84-C-0006

FIRST SEMI-ANNUAL REPORT (1st periodic report)

Report period: 1st March, 1984 - 31st August, 1984

The research reported in this document has been made possible through the support and sponsorship of the U.S. Government through its European Research Office of the U.S. Army. This report is intended only for the internal management use of the Contractor and the U.S. Government.

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Scientific work done

The contractor's technical proposal described the ultra-high vacuum mechanical microprobe recently installed in our laboratory, and designed to investigate sliding contact at the "single asperity" level. The project started on March 1st, 1984, since when we have appointed a post-doctoral research associate, Dr. J.D.J. Ross, who will work on this project for 2½ years (from 1st June 1984, the date of his appointment), and whose salary is paid from the funds awarded. Dr. Ross worked with Dr. C.R. Brookes at the University of Exeter on size effects in hardness, deformation in diamond cubic solids, and the effects of sliding contact on the microhardness of bearing steels. Each feature of the microprobe has been made to function successfully with the help of certain specially-fabricated additional components. These features include the microprobe assembly itself, the ultra-smooth rigid sliding contact system, the field emission assembly, and the electron and ion beam systems.

As described in the supplement (dated Oct. 21, 1982) to the technical proposal, the project also involves complementary work on the measurement of microhardness at sub-micron depths. The special microindentation instrument needed for this research was used by the principal investigator during the first three months of the project to continue in particular with the study of time-dependent effects in the indentation of nickel and type 304 steel: it is perhaps surprising that such effects are significant even at ambient temperature, when the indentation depth is less than ca. 0.5 µm. With the help of our Paris colleagues Dr. D. Maugis and Dr. M. Barquins, and with specimens provided by Dr. I.L. Singer of the Naval Research Laboratory, Washington and by Dr. J - C Pivin of the University of Paris - Sud (Orsay, France), we have been able to draw certain conclusions regarding the characterisation of sub-micron surface layers by indentation:

1. Materials may be characterised at depths in the 10-300 nm range, through measurements of plasticity indices, elastic recovery parameter, elastic modulus, and indentation creep parameters.
2. Significant low-temperature indentation creep at small depths can occur in metals even at ambient temperature. When this is so, attempting to derive hardness values is of little use. Instead, it is more profitable to measure empirical creep parameters such as characteristic time and depth.
3. The method of continuous depth recording is simple and rapid compared with methods involving measurements of indent diameter. This applies to both experiment and theoretical interpretation especially when elastic recovery or indentation creep is observed.

4. The effect of ion implantation upon creep behaviour can be as important as its effect upon the hardness itself. For example, in nickel and type 304 steel it can eliminate the "accelerated creep" which otherwise appears when the indentation depth reaches 100-300 nm.

5. The plasticity indices may be related to macroscopic hardness values, and the creep parameters to activation energy and athermal yield stress, if we make certain simplifying assumptions regarding full plasticity and low-temperature creep mechanisms. Both experimental work and the required theory are at an early state, especially where surface or scale effects are involved or where the yield stress varies with depth.

This work was presented at the I.M.S./A.S.T.M. Microindentation Hardness Testing Symposium and Workshop in Philadelphia, 15-18 July, 1984. During the same visit to the U.S.A. the principal investigator had useful discussions with Dr. P. Sagalyn of A.M.M.R.C., Watertown, Dr. C.J. McHargue and Dr. W.C. Oliver of O.R.N.L., Oak Ridge, and Dr. I.L. Singer of N.R.L., Washington.

Research plans for the remainder of the contract period

The sliding contact experiments in vacuum are expected to begin shortly, and their aim remains very much ^{as} outlined in the original proposal. The microindentation experiments are continuing, and both types of experiment will involve the same materials: at present, nickel in the form of (a) single crystal, (b) polycrystalline (unimplanted and implanted with boron and phosphorus), (c) vapor-deposited film, and (d) glassy Ni-B alloys.

Personnel; conferences etc

The appointment of the research associate, and the paper presented at the I.M.S./A.S.T.M. symposium, have been mentioned above.

Unused funds remaining at 31st August, 1984:

\$ 46,565

Important property acquired with contract funds:

None.



H.M. Pollack

A-1

Title: FINE-SCALE MECHANICAL PROPERTIES OF SLIDING SOLIDS

Principal investigator: Dr. H.M. Pollock, Department of Physics

Contractor: University of Lancaster (attn. Finance Officer),
Lancaster, England LA1 4YW

Contract number: DAJA 45-84-C-0006

SECOND PERIODIC REPORT

Report period: 1st September, 1984 - 28th February, 1985

The research reported in this document has been made possible through the support and sponsorship of the U.S. Government through its European Research Office of the U.S. Army. This report is intended only for the internal management use of the Contractor and the U.S. Government.

87-9-4-025

1. Scientific work done

The contractor's technical proposal described the ultra-high vacuum mechanical microprobe designed to investigate sliding contact at the "single-asperity" level. Using a fine pointed metal tip in contact with a nickel crystal at very low or even negative loads we aim to measure the static and sliding friction forces at the interface. At the same time, changes in real area of contact are monitored by means of electrical resistance measurements, and any transfer of material on separation is monitored through measurement of field electron emission characteristics. Preliminary experiments show that the required slow and stable sliding speeds can be achieved, and adhesion (negative loads) can be measured with enough sensitivity. At present we are evaluating the relative merits of two alternative methods of measuring the frictional force : an optical lever and a capacitance technique.

The complementary work on measurement of microindentation hardness and creep at submicron depths, as described in the first periodic report, has made rapid progress. Through the collaboration with our colleagues at Orsay (France), Nickel-Boron films have been prepared by (a) ion implantation and (b) chemical deposition. The following surface-mechanical properties have been measured: friction and wear (at Orsay) and surface hardness and creep-resistance (at Lancaster). We have to some extent been able to clarify how far these properties depend on radiation damage, on solute hardening resulting from increasing boron concentrations, on the amorphization seen above a concentration threshold, and on recrystallization due to annealing. The principal difference between the dry friction behaviour of the amorphous films, compared with Ni or crystalline Ni_3B , seems to be an improvement of the seizure-resistance, which is correlated with the lower reactivity of the glassy compound, and also with its high surface hardness and creep-resistance. However these last two properties depend more strongly upon the boron concentration itself and on radiation damage, than on the degree of amorphization. For example, a dramatic change in indentation creep properties is seen for boron concentrations in the range 1% to 5%: at such concentrations, implanted ions cause lattice swelling and disorder. For concentrations in the range 5% to 14% (formation of amorphous clusters), the creep-resistance increases more slowly, finally levelling off at boron concentrations in the range 14% to 28%, corresponding to amorphous fractions between 50% and 100%. Some of this work will be presented at the EUROTRIB 85 congress at Lyons in September 1985, and two papers are in preparation.

2. Research plans for the remainder of the contract period

Especially in view of the information on seizure-resistance of Ni-B specimens first priority at present is to begin the planned complementary adhesion and

sliding contact experiments using the ultra-high vacuum mechanical microprobe. Both these and the continuing microindentation experiments will involve the same specimen materials, as outlined in the original proposal.

3. Conferences

The planned EUROTRIB 85 conference paper mentioned above is entitled "Rheology and dry friction of amorphous and crystalline NiB films" by J. Takadoum et al.

4.

5. Annex (Please see attached sheet)

Dr. J.D. Ross  p.p. Dr. H.M. Pollock.

28th February, 1985

5. Annex to second periodic report on Contract No. DAJA 45-84-C-0006.

(a) Unused funds remaining at 28th February, 1985:

\$ 34,767.

(b) Important property acquired with contract funds:

None.

Title: FINE-SCALE MECHANICAL PROPERTIES OF SLIDING SOLIDS

Principal investigator: Dr. H.M. Pollock, Department of Physics

Contractor: University of Lancaster (attn. Finance Officer),
Lancaster, England LA1 4YW

Contract Number: DAJA 45-84-C-0006

THIRD PERIODIC REPORT (2nd semi-annual report)

Report period: 1st March, 1985 - 31st August, 1985

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SECTION II - PERIODIC REPORT

This Report is a major means whereby the Scientific Officer and the Contracting Officer are kept informed of the current status of the Contract.

A. Cover or Title Page - will include the following:-

- (1) Title of Research Project
- (2) Name of Principal Investigator
- (3) Name of the Contractor
- (4) Contract Number
- (5) The words " Periodic Report". (In blank space insert appropriate report number, for example: 1st, 2nd, 3rd).
- (6) Month and year of start and end of report period, example May 1979 - July 1979.
- (7) The following statement: "The Research reported in this document has been made possible through the support and sponsorship of the U.S. Government through its European Research Office of the U.S. Army. This report is intended only for the internal management use of the Contractor and the U.S. Government".

B. Body of the Report

Research data obtained during the total research period must be included in the Final (Annual) Technical Report. Therefore, detailed data is not required in the Periodic Reports. However, the Contractor may include in the Periodic Report any specific data he wishes to bring immediately to the attention of the European Research Office of the U.S. Army. The Periodic Reports will include the following:-

- (1) A statement in approximately 500 words of the Scientific Work done during the reporting period. Include results of attendance at scientific meetings and list of those papers presented or submitted for publication to which at least partial contractual sponsorship must be attributed.
- (2) A brief statement of research plans for remainder of the contract period.
- (3) A statement of significant administrative actions during the period reported, such as personnel changes, important conferences and the like.
- (4) Any other information considered important by the Contractor.
- (5) Annex (to be attached to report):-
 - a. A statement showing the amount of unused funds remaining on the contract at the end of the period covered by the report.
 - b. A list of important property acquired with contract funds during this period.

1. Scientific work done

As described in the two previous reports, the chief aim of the research is being pursued with the help of two different approaches. One of these involves the study of sliding contact between similar or dissimilar metals at the "single-asperity" level, in controlled environments starting with ultra-high vacuum. We have completed the evaluation, mentioned in the second report, of the two alternative merits of measuring the microneutron-level frictional forces involved : a system based on an optical lever has now been incorporated into the equipment, and friction traces are stored on floppy disc via a computer program. So far, experiments have been carried out in ambient atmosphere only. Although these could usefully be correlated with micro-indentation tests performed in air (see below), subsequent and potentially more reproducible and valuable experiments will be carried out in U.H.V. under conditions where the test surfaces will be thoroughly cleaned and characterized, and any changes in surface geometry or transfer of material at the nanometre level will be monitored.

Our other approach involves measurement of microindent ion hardness and creep at sub-micron depths. The previous report summarized work on nickel-boron films prepared by (a) ion implantation and (b) chemical deposition, in collaboration with our colleagues at the Orsay Centre for Nuclear Spectrometry and Mass Spectrometry (CSNSM), France: the poster presentation to be presented at the EUROTRIB 85 congress at Lyons (later this month) will describe the effect of boron concentration, radiation damage and degree of amorphisation upon seizure-resistance, surface hardness and creep resistance, as summarized in February 1985. Since then we have carried out a large number of further tests in order to fill in important details, and two papers have been written for submission to journals in the near future. (This has been possible thanks to an eight-week visit to Lancaster by J. Takadoum from Orsay in order to help with experiments, and two short visits to France by H.M. Pollock). The first paper is entitled "Limits to the hardness testing of films thinner than a nanometre" (J.D.J. Ross, J. Takadoum, J.C. Pivin and H.M. Pollock) and is to be submitted to the journal Thin Solid Films. We conclude that for layers of thickness ca. 100-600 nm, the method of continuous indentation depth recording may be used to obtain a quantitative mechanical characterization of a film material, over a large range of depth within the film. This conclusion applies to both soft and hard layers. The effect of the substrate often appears to be felt at a film thickness/indentation depth ratio significantly different to that predicted by macroscopic indentation theory: for the thinnest of the soft films studied, this ratio tended to be greater than expected, while for the hard layers the opposite was found. The second paper is entitled

"The mechanical properties of boron and phosphorus-implanted nickel, discussed in terms of increasing disorder and amorphicity" by J. Takadoum, J.C. Pivin, H.M. Pollock and J.D.J. Ross, and discusses in detail the work on surface hardness and creep-resistance described in the EUROTRIB poster presentation. Related experiments on chemically-grown Ni-B films have been done also, in collaboration with Ecole Nationale Supérieure of Metallurgy and Micro-techniques (E.N.S.M.M.), Besançon, France. This collaboration has involved further visits in both directions.

2. Research plans for the remainder of the contract period

Our first priority remains the planned complementary adhesion and sliding contact experiments using the ultra-high vacuum mechanical microprobe. Using the same specimen materials, as outlined in the original proposal, we will also continue to use the microindentation equipment to study the phenomenon of indentation creep in greater detail, beginning with creep experiments in late September/October 1985 in which we will have the assistance of a visitor (Dr. T. Zajíček) from the laboratory just mentioned (E.N.S.M.M.).

3. -

4. -

5. Annex Please see attached sheet.

H.M. Pollock

1 September 1985

5. Annex to third periodic report on Contract No. DAJA 45-84-C-0006:

(a) Unused funds remaining at 31st August 1985:

\$56,565 less (10,000 + 11,798 + 10,000) = \$24,767

(b) Important property acquired with contract funds: None.

Title: FINE-SCALE MECHANICAL PROPERTIES OF SLIDING SOLIDS

Principal investigator: Dr. H.M. Pollock, Department of Physics

Contractor: University of Lancaster (attn. Finance Officer),
Lancaster, England LA1 4YW

Contract Number: DAJA 45-84-C-0006

FOURTH PERIODIC REPORT (2nd annual report)

Report period: 1st September 1985 - 28th February, 1986

The research reported in this document has been made possible through the support and sponsorship of the U.S. Government through its European Research Office of the U.S. Army. This report is intended only for the internal management use of the Contractor and the U.S. Government.

erosion-cavitation, or initiation of cracks in fatigue is due in fact to a change of structure. Ni-B glasses are hard but fully plastic. The strain rate does not seem to be an important factor in the change of surface mechanical properties with amorphization. Thus, a one-to-one relationship cannot be established between the amorphous or crystalline nature of surfaces and their mechanical properties, since disordered crystalline surfaces can be as hard as amorphous ones; the chemical nature of the addition must also be considered. However the changes of hardness observed after a relaxation or crystallisation annealing seem to be consistent with the hypothesis put forward concerning the deformation mechanisms of glasses and their expected tribological performances. A random distribution of atoms in supersaturated solution, or a high density of extended defects induced by irradiation, or the presence of clusters, appear also to suppress the scale effect commonly observed in indentation of metallic surfaces.

The important and related subject of room-temperature indentation creep has been pursued primarily in connection with specimens supplied by Dr. I.L. Singer of the Naval Research Laboratory. We have completed further measurements on his samples of type 304 steel, parts of which have been transformed to martensite by polishing, and/or implanted with nitrogen ions. We believe that the time-dependent fine-scale indentation behaviour, studied at depths as small as 50 nanometres, is a factor in determining the abrasive wear-resistance of the material. At this level its behaviour may often be described in terms of an effective viscosity : whereas strain-hardening has a negligible effect upon the viscosity, ion implantation increases its value dramatically. On this subject we are involved in discussions with members of Professor M.F. Ashby's group at Cambridge University's Engineering department, and a paper is being prepared for publication.

2. Research plans for the remainder of the contract period

Having established that frictional forces can be measured at the scale of these experiments, our aim is to explore further the relationships between static and sliding friction, contact resistance and adhesion in order to distinguish between discrete mechanisms of friction. We intend to study these parameters on well characterised surfaces (analysed by Auger emission spectroscopy) covering a range of conditions i.e. from "clean" to heavily contaminated. In addition to tests on pure metals we shall be investigating the frictional characteristics of ion implanted discs in conjunction with the low load indentation experiments described above.

3. -

4. -

5. Annex Please see attached sheet.

5. Annex to fourth periodic report on Contract no. DAJA 45-84-C-0006:

(a) Unused funds remaining at 28th February 1986:

\$ 56,565 less (10,000 + 11,798 + 10,000 + 12,821)

= \$ 11,946.

(b) Important property acquired with contract funds: None.

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