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EXPLORATORY DEVELOPMENT ON THE HYDROGEN EMBRITTLEMENT OF HIGH STRENGTH STEEL DURING MACHINING

BOEING AEROSPACE COMPANY

PREPARED FOR

AIR FORCE MATERIALS LABORATORY

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This experimental program was undertaken to investigate the possibility of machining fluids being a source of hydrogen for a given high strength aerospace structural alloy. Unexpected catastrophic failure of high strength steel structures can occur as a result of hydrogen embrittlement if the material absorbs high levels of hydrogen during fabrication or when the hardware is in use. Test specimens made of AISI 4340 high strength steel (heat treated to 260,000 - 280,000 lb/in^2 strength level) of known hydrogen concentration were subjected to a specified schedule of gentle and abusive milling and grinding operations using different machining fluids. Following the machining operations the specimens were analyzed for excess hydrogen above the base level with a Boeing developed "Ultrasensitive Hydrogen Analysis System". A total of six different machining fluids with different active chemical components were used in this study. Experimental results are presented with a statistical analysis of the hydrogen concentration data.

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FOREWORD

This report was prepared by The Boeing Aerospace Company, Seattle, Washington, under Air Force Contract F33615-73-C-5120, "Exploratory Development on Hydrogen Embrittlement of High-Strength Steels During Machining". The contract was initiated by the Air Force Materials Laboratory under Project No. 7312, Task No. 731201. The project scientist monitoring this program was Captain Phillip A. Parrish, AFML/LLP.

This report covers work performed during the period of 1 April 1973 through 30 June 1973 and was submitted by the author in July 1973. This report is also released as Boeing Document D180-17582-1 for internal control at The Boeing Company.

This program was conducted by the Research and Engineering Division of The Boeing Aerospace Company, Seattle, Washington, under the supervision of H. W. Klopfenstein, Structures Research and Development Manager. The Program Leader was Mr. G. E. Hughes, Supervisor, Materials and Processes Group and the Technical Leader was Dr. K. B. Das. Mr. J. F. Bruggeman, from Manufacturing Technology group, monitored test specimen fabrication. Hydrogen analysis was conducted by K. R. Torluemke and the specimen grinding was done by R. Gossett under the supervision of H. Hansen, V. Knott and R. Peterson.

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ABSTRACT

This experimental program was undertaken to investigate the possibility of machining fluids being a source of hydrogen for a given strength aerospace structural alloy. Unexpected catastrophic failure of high strength steel structures can occur as a result of hydrogen embrittlement if the material absorbs high levels of hydrogen during fabrication or when the hardware is in use. Test specimens made of AISI 4340 high strength steel (heat treated to 260,000 - 280,000 lb/in^2 strength level) of known hydrogen concentration were subjected to a specified schedule of gentle and abusive milling and grinding operations using different machining fluids. Following the machining operation the specimens were analyzed for excess hydrogen above the base level with a Boeing developed "Ultrasensitive Hydrogen Analysis System". A total of six different machining fluids with different active chemical components were used in this study. Experimental results are presented with a statistical analysis of the hydrogen concentration data.

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1.0 INTRODUCTION

Hydrogen as a causative agent in adversely affecting the mechanical properties of high strength aerospace structural materials, especially high strength steels, is well known[1]. Stress corrosion cracking in titanium and other transition metal alloys has been attributed to hydrogen[2] and porosity in aluminum welds due to hydrogen is well documented[3]. Once hydrogen enters high strength steels these metals can lose their ductility and depending on the level of hydrogen can suffer brittle failure when subjected to stress. This much studied but still vaguely understood process is known as "hydrogen embrittlement". In some cases presence of only trace amounts of hydrogen will greatly reduce the tolerable stress levels below those which a high strength steel structure is initially designed to withstand, leading at times to unexpected catastrophic failure as a result of hydrogen embrittlement. The exact concentration of hydrogen which may be deleterious to the strength of a steel structure is not clearly known, although the published literature[1] suggests the amount to be very small. Also described in the literature[1] is the fact that the resistance of statically loaded, hydrogen containing high strength steel to delayed brittle failure decreases rapidly with increasing strength-level[4] after a limiting value has been exceeded. The practical importance of this phenomenon is vividly illustrated when one looks at the ever increasing demand for steels of higher and higher strength levels for weight saving in the aerospace industries.

2.0 GENERAL BACKGROUND AND OBJECTIVE OF THE PROGRAM

Hydrogen may be absorbed by metals both during processing and when the finished products are in use. Certain operations in the fabrication of ferrous alloys which are particularly prone to the introduction of hydrogen include heat treatment, hot working, welding, chemical milling or pickling, and electroplating. In general, any process producing atomic hydrogen on the surface of a metal can introduce hydrogen into the metal. Some 15 years ago the hydrogen embrittlement phenomenon became a matter of serious concern because of the increasing frequency of

of brittle failures of electroplated landing gears, cadmium plated steel fasteners, and weaponry. This problem still exists today in spite of improved quality control of fabrication operations. Comparatively little work has been done in the domain of correlating hydrogen content to hydrogen embrittlement. Very little is known about the relationship between the distribution of hydrogen inside the metal and its embrittlement behavior. This is because of the technological difficulties in accurately measuring the hydrogen content in the low parts per million range. As a result of this measurement problem industries have tried to identify and eliminate all the possible sources of hydrogen in an attempt to reduce the susceptibility to brittle failures of hardwares.

The Boeing Aerospace Company's strong interest in maintaining adequate cost effective hydrogen detection capability for controlling and further studying this vital problem of hydrogen embrittlement has been demonstrated by the continuation of development studies during the past several years. Such studies have led to the development of a unique "Ultrasensitive Hydrogen Analysis System" which can detect hydrogen concentrations of as low as a few parts per billion in a carrier gas stream. High detection sensitivity and the use of a unique calibration standard gives accurate results in the low parts per million range for the bulk hydrogen analysis of a material.

Among the list of sources that have been identified machining fluids being a possible significant source (during a specific machining operation) is missing. For example, two of the most important functions of a grinding fluid are cooling and lubrication. However, the heat generated during a typical grinding operation could often be sufficient to breakdown the hydrocarbon based grinding fluid thereby providing a source of nascent hydrogen on the grinding wheel-workpiece interface. The theoretical possibility of machining fluids as a hydrogen source, the lack of knowledge of dangerous hydrogen levels and the timely development of the uniquely sensitive and cost-effective Boeing's Hydrogen Analysis System" precipitated into this study program.

The objective of this study program was to determine if chemical reactions with machining fluids during a preselected machining operation can be a significant source of entrapped hydrogen. Test specimens made of AISI 4340 high strength steel (heat treated to 260,000 - 280,000 lb/in^2 strength level) of known hydrogen concentration were subjected to a specified schedule of gentle and abusive milling and grinding operations using different machining fluids. Following machining the specimens were analyzed for excess hydrogen above the base level with the ultrasensitive "Hydrogen Analysis System". A total of three different oil base and three water miscible oil emulsion machining fluids, composed of different active chemical components, were used in this study. Experimental results are presented with a statistical analysis of the hydrogen concentration data.

3.0 EXPERIMENTAL

3.1 HYDROGEN ANALYSIS SYSTEM

A schematic of the hydrogen detector is shown in Figure 1. The system essentially consists of an airtight specimen holder which is placed inside the work coil of an induction heater for the purpose of extracting hydrogen from the test specimen, a micro-quantitative gas metering system or MGM system, a semi-permeable membrane (from here on referred to as SPM), a vac-ion pump, and a source of high purity argon gas. Hydrogen present as water, organic material as well as elemental, is released from a metal by heating the metal. The hydrogen thus liberated is directed toward the activated SPM by the flowing argon gas (carrier gas). The SPM lets only hydrogen permeate through it while remaining impermeable to other gases. On the other side of the SPM is a continuously pumped high vacuum chamber (ion-pump housing). Thus the SPM has a high vacuum on one side and about one atmosphere pressure on the other.

The pressure in the ion-pump chamber is measured by monitoring the ion-current through the pump. Once the chamber is pumped down to its base vacuum, any permeation of hydrogen through the SPM will

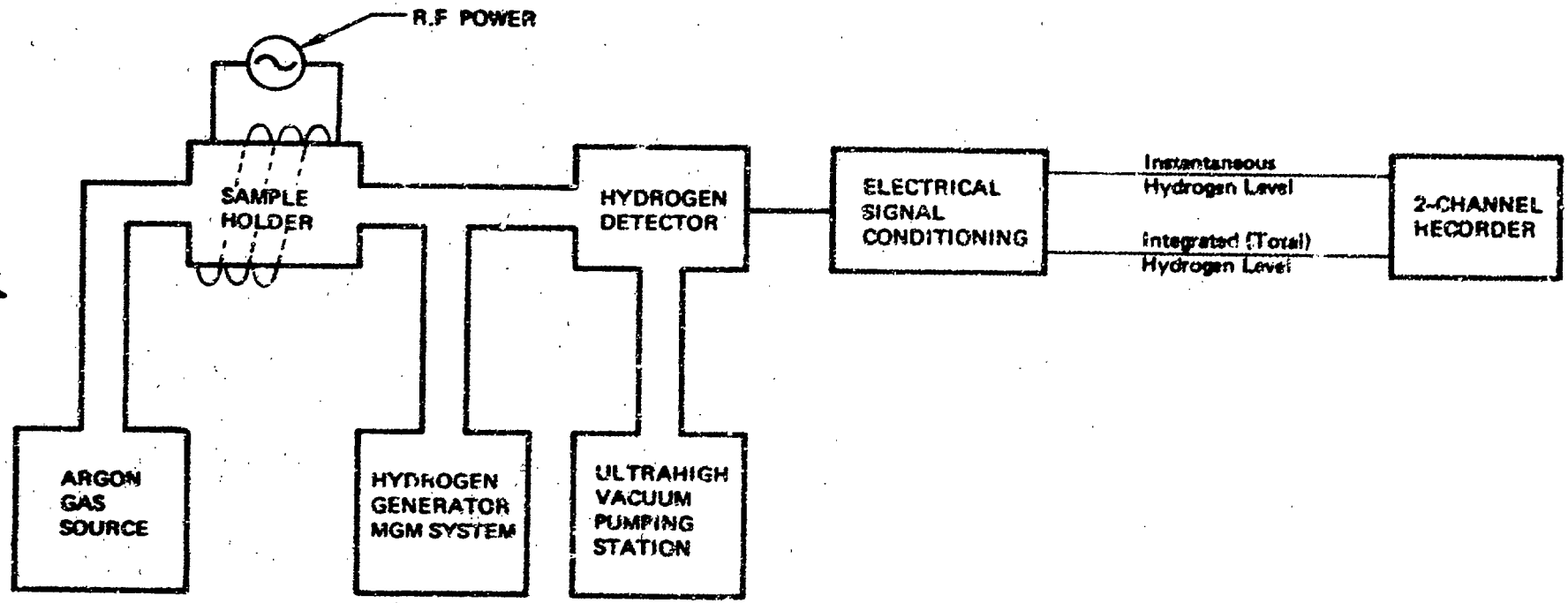

Figure 1: SCHEMATIC OF HYDROGEN DETECTOR

result in an increase in the ion-current reading as seen by the ion-pump. An increase in the ion-current reading is therefore directly proportional to the amount of hydrogen in the carrier gas stream. Thus, if the detector readout is calibrated by introducing known amounts of hydrogen in the vacuum system, thereby establishing a relationship between the concentration of hydrogen in the gas stream and the ion-current readings, one can easily estimate the amount of hydrogen in the gas stream from an unknown source. In the present case the detector is calibrated by introducing known amounts of hydrogen in the vacuum chamber by the MGM system, and a relationship is established between the concentration of hydrogen in the gas stream and the ion-current readings. Such a calibration procedure also provides a check for the linearity of the detector and gives a measure of its sensitivity. The sensitivity of the detector depends upon the flow-rate of the carrier gas, and the temperature of the SPM. Thus, in order to maintain the same permeation efficiency for hydrogen from the MGM system and the test specimen it is essential to keep these parameters constant.

For bulk hydrogen analysis the experimental approach essentially consists of extracting hydrogen from the test specimens using an induction furnace and then measuring the amount of hydrogen thus extracted with the hydrogen detector. Hydrogen, after permeating through the SPM, arrives in the detector housing where it is detected by an appropriate sensing element and an instantaneous electrical signal representative of the amount of hydrogen present is recorded. In practice when the specimen is heated the time for complete extraction of hydrogen depends upon the rate at which hydrogen diffuses out of the specimen. This rate of diffusion is proportional to the temperature of the specimen and as such the time required for complete extraction of hydrogen depends on how rapidly the temperature of the specimen is raised. In the case of bulk hydrogen analysis the instantaneous hydrogen signal generally traces out a Maxwellian type of curve representing complete extraction of hydrogen from the test specimen. When this curve is integrated electronically it results in an S-shaped curve giving a direct numerical value for the area under the Maxwellian curve.

The design of the system is such that not all the hydrogen directed toward the SPM goes through the detector. Moreover, the diffusion of hydrogen through SPM itself is a time dependent process. When using an induction furnace such factors are eliminated by following a special operating technique. Here the sample is heated to its melting point in a programmed manner and held at this temperature until complete extraction of hydrogen is accomplished. Both the instantaneous and integrated hydrogen signals are recorded as a function of time. Following the hydrogen extraction, at the same carrier gas flow-rate $(dv/dt)_{Ar}$, a known amount of hydrogen is introduced into the gas stream from the MGM system for a precisely known time Δt. The MGM system consists of a pinched capillary tubing with one of its ends connected to the carrier gas channel and the other connected to a high purity hydrogen bottle, a low pressure gas regulator, and gas bleed-off system. By regulating the hydrogen gas pressure, varying amounts of hydrogen can be introduced into the carrier gas stream. The rate of hydrogen gas flow through the pinched capillary is determined by connecting it to a glass capillary tubing (of precisely known dimensions) containing a little slug of mercury. From the knowledge of the area of cross-section of the glass capillary and the time taken for the mercury column to move a given distance, the rate $(dV/dt)_H$ is computed. The ratio of $(dV/dt)_H$ and $(dV/dt)_{Ar}$ then gives a measure of ppm of hydrogen in the carrier gas stream. It should be noted that $(dV/dt)_H \approx 10 cc^{-4}/_{min}$ is much smaller than $(dV/dt)_{Ar} \approx 100 cc/_{min}$. Furthermore, the product of $(dV/dt)_H$, Δt, and density of hydrogen "ρ"$_H$ gives the amount of hydrogen M, in grams, introduced into the gas stream in time Δt. The units of the integrated signal is coulombs. Thus if the integrated signal from the known hydrogen input M_1 (grams) and the test sample is Q_1 and Q_2 coulombs respectively, the amount of hydrogen M_2 (grams) from the test sample can be calculated from the relation $M_2 = (Q_2/Q_1)M_1$.

To further illustrate the method of analysis an example of a (certified) NBS sample is given here which had a known hydrogen concentration of 32 $\pm$ 2 ppm. After pumping the detector housing down to its base level a carrier gas flow-rate of 50 cc/min was chosen for the experiment.

Thereafter, hydrogen was extracted from the NBS sample by heating the specimen in the induction furnace. The time for complete extraction from this sample, which weighed 0.1847 gms, was about 16 minutes. The integrated hydrogen signal gave a value of 190400 microcoulombs. Thereafter, by appropriately regulating the hydrogen gas pressure on the MGM system and maintaining the same carrier gas flow-rate of 50 cc/min, hydrogen was introduced into the argon gas stream at the rate of 19.05×10^{-3} cc/min for 4.0 minutes. In other words, in 4 minutes, 6.38×10^{-6} grams of hydrogen was introduced into the gas stream. This gave an integrated hydrogen signal of 207000 microcoulombs. Using the relation

$$\frac{207000 \times 10^{-6} \text{ ccul}}{5.38 \times 10^{-6}} = \frac{190400 \times 10^{-6} \text{ coul}}{X \text{ grams}}$$

$$\text{or} \quad X = 5.869 \times 10^{-6} \text{ grams},$$

the total bulk hydrogen in the NBS sample was found to be 5.869×10^{-6} grams. Dividing X by the weight of the sample gives the level of hydrogen in ppm by weight which in this case was 31.78. This value, as can be seen, is well within the uncertainty limits of the NBS sample.

In the present program the test specimens were analyzed for hydrogen content in an identical manner as described above for the NBS specimen. A typical curve for the hydrogen analysis of a NBS standard containing 215 $\pm$ 6 ppm hydrogen is shown in Figure 2. During the course of this experimental program certified NBS samples containing 32 $\pm$ 2 ppm or 98 $\pm$ 5 ppm of hydrogen were analyzed at a schedule of one per week to cross-check the detector's performance and accuracy.

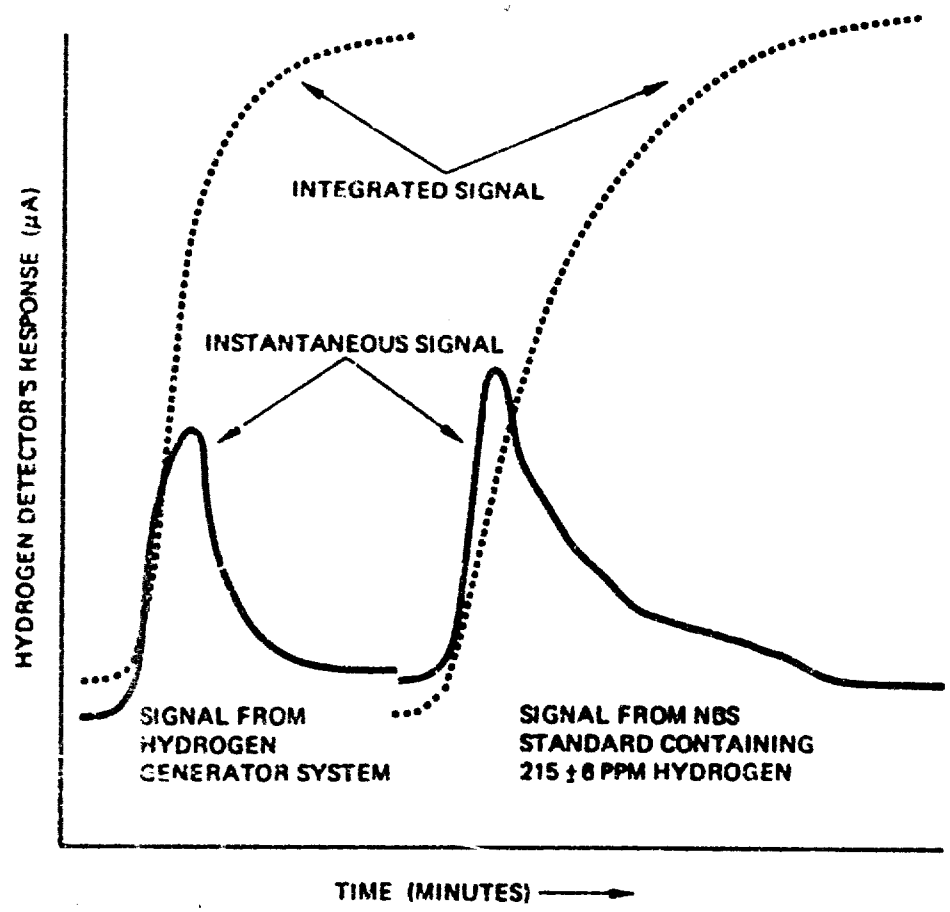

Figure 2: HYDROGEN ANALYSIS OF NBS STANDARD NO. 354 CONTAINING 215 ± 6 PPM HYDROGEN

3.2 TECHNICAL APPROACH

The technical approach essentially consisted of subjecting test specimens made out of AISI 4340 high strength steel of known hydrogen concentration, to a specified schedule of gentle and abusive milling and grinding operations, using machining fluids of different active chemical components (neutral, chlorinated and sulfurized) typical of commercial fluids. The machined specimens were then analyzed for excess hydrogen with the ultrasensitive hydrogen analysis system. The sequence of events is shown in the events logic diagram, Figure 3.

3.2.1 Preparation of Base or Starting Material

The AISI 4340 steel stock material (ordered per requirements of AMS specification 6359A) was 3/8" thick and 6' x 6' in size. Specimens for hydrogen analyses were approximately cubical in shape (1/4" x 1/4" x 1/4"). These dimensions were determined by furnace allowances, such as diameter of the quartz tubing and work coil requirements of the induction heater.

The base material was prepared by end milling the as-received sheet stock material (prior to heat treatment) as per the schedule outlined in item A.1 of Table II. End milling was carried out to reduce as-received material thickness to about .030 in. over the actual size of the final finished test specimens. The material in this milled condition is referred as base material in the following sections. Both sides of the specimens were machined. Final thickness of all test specimens were 0.25 inches.

3.2.2 Hydrogen Analysis of Base Material

A three inch long and 1/4 inch wide section was obtained from the base material prepared in 3.2.1. Both sides of this piece was milled as per item A.1 of Table II to its final thickness of 0.25". This final milled piece was then heat treated by austenizing at

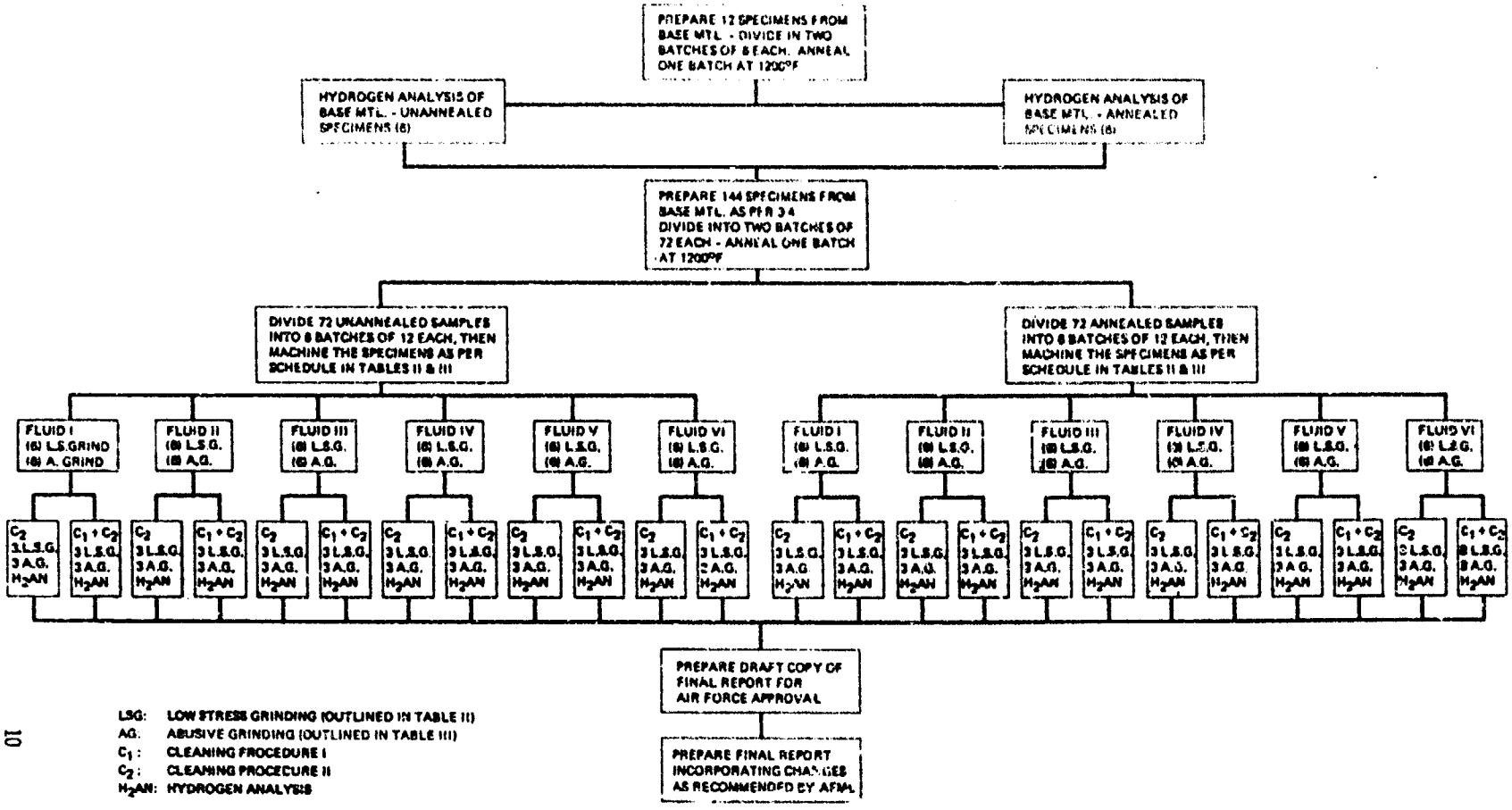

Figure 3: EVENTS LOGIC DIAGRAM

1550°F for one (1) hour and oil quenching, followed by tempering at 425°F for two (2) hours to obtain 260,000 - 280,000 lb/in^2 tensile strength condition.

Thereafter the sequence of events proceeded as follows:

a. Twelve (12) samples about 0.25" L x 0.25" W in size were cut with a clean carbide blade.

b. All the samples were then cleaned (soon after cutting the specimen adequate time was given for cooling and the specimens were always handled with forceps) as per cleaning procedure C_1 shown below:

   - Wash with acetone
   - Wash with carbon tetrachloride
   - Wash with pure Freon 113 (distilled and stored in steel container)

c. After the above cleaning cycle, the specimens were thoroughly dried and then weighed carefully. From here on, the samples were always stored in a clean, dry dessicator.

d. The above set of twelve samples was then divided into two batches of six each. One batch of six samples was annealed at 1200°F for three hours in a vacuum furnace and furnace cooled. The specimens were stored in a dessicator after the heat treatment.

e. The remaining six unannealed samples were analyzed for hydrogen concentration. A statistical analysis of the concentration data was conducted to determine the standard deviation, and to express the random variation from the average value at a 90% confidence level.

f. The annealed specimens were then analyzed for hydrogen content and the results expressed as in 3.2.2(e).

3.3 MACHINING FLUIDS

Six model machining fluids were chosen for this study program. These represent the numerous and different machining fluids commonly used in machining. The compositions of these fluids are provided in Table 1.

They include:

- Oil Base Cutting Fluids
  - Neutral
  - Chlorinated
  - Sulfurized
- Soluble Oil Emulsions
  - Neutral
  - Chlorinated
  - Sulfurized

3.4 HYDROGEN ANALYSIS OF SPECIMENS SUBJECTED TO LOW STRESS GRINDING OPERATIONS

For this phase of the study an appropriate section from the base material (as prepared in item 3.2.1) was obtained which was sufficient for preparing 72 samples of about 1/4" x 1/4" x 1/4" in final size. The cut section was heat treated as per procedure outlined in 3.2.2. Subsequent to heat treatment, a batch of 72 samples was prepared as per 3.3.2(a) through 3.2.2(c). After dividing the samples into two batches (36 in each batch), one of the batches was annealed at 1200°F for three hours as in 3.2.2(d). The other portion of the base material was saved for abusive grinding study (3.5). Thereafter the analysis proceeded as follows:

3.4.1 Unannealed Specimens

The samples were divided into groups of six each. A predetermined amount of material was machined by the machining method detailed under Sections A.2 and B (grinding after heat treatment and low stress grinding conditions - final cuts) of Table II using one of the prescribed machining fluids as described in 3.3. (Both sides of the specimens were machined). After machining, all the specimens were cleaned by the cleaning procedure C_2 as shown below:

- Agitate specimen in naphtha for five minutes - drain.
- Immerse in boiling solution of alkaline cleaner (500cc water + 32 gms of KELITE 235) for 10 minutes.
- Rinse in hot distilled water.
- Successively run in distilled water until no residue alkali is detected.
- Blow clean with filtered dry air.

a. Thereafter, three out of six specimens were analyzed for hydrogen concentration. The remaining three specimens were analyzed for hydrogen after following cleaning and storage procedure C_1 (see 3.2.2(b)). Such an analysis gave the following information:

Amount of hydrogen introduced (if any) in 4340 steel having a known base level of hydrogen during a gentle machining operation as described in Table II using a specific model machining fluid.

b. The above procedure (3.4.1(a)) was repeated with the remaining five groups of specimens using the same machining techniques (Table II:A(2) and (B)), using each of the other machining fluids specified in 3.3 and analyzing for hydrogen concentration.

3.4.2 Annealed Specimens

Annealing the cut samples at an elevated temperature in a vacuum furnace should remove most of the hydrogen from the specimens. Furthermore, the cutting operation leaves the specimens in a highly stressed state with a large density of dislocations. A large density of dislocations implies an open structure. Literature shows[5,6] that such a specimen is liable to absorb more hydrogen when subjected to hydrogen atmosphere than one which has a much smaller density of dislocations or a relatively closed structure. The above annealing operation should therefore provide specimens with much smaller density of dislocations in their starting material compared to their unannealed counterparts.

All the annealed specimens were subjected to a similar analysis as outlined in 3.4.1.

3.5 HYDROGEN ANALYSIS OF SPECIMENS SUBJECTED TO ABUSIVE GRINDING OPERATIONS

From the remaining portion of base material used in 3.4 a batch of 72 samples was prepared as per 3.2.2(a) thru 3.2.2(c). Thereafter the entire procedure as outlined in Section 3.4 was repeated using the abusive machining technique described in Table III for each of the prescribed model machining fluids as given in 3.3.

3.6 VACUUM ANNEALING, MILLING AND GRINDING OPERATIONS

Subsequent to milling the specimens were heat treated to 260,000 - 280,000 lb/in^2 strength level and then divided into two batches. One batch was annealed for 3 hours at 1200°F at a vacuum level of 1×10^{-6} to 5×10^{-6} Torr and furnace cooled in a turbo-molecular pumped, liquid nitrogen trapped Brew vacuum furnace. The samples were marked U or A to identify their unannealed or annealed condition. All the specimens were then subjected to either gentle or abusive grinding (using one of the machining fluids at a time) as per the schedule outlined in Tables II and III prior to the hydrogen analysis. As can be seen from the events logic diagram, Figure 3-3, a total of 24 specimens are required for hydrogen analysis for each machining fluid. However, since the hydrogen analysis is a destructive test additional samples are necessary at times to verify a questionable run. As such 8 additional samples (1 extra sample for each set of 3) were prepared along with each batch of 24 so that six reliable readings per sequence of low stress or abusive grinding (with two different cleaning procedures) and unannealed or annealed condition can be obtained for statistical analysis. Soon after grinding, the low stress and abusively ground specimens were transferred to appropriately marked containers which contained a small amount of the same mixture of machining fluid as that used during the grinding operation. After bringing the specimens from the shop to the hydrogen lab the samples were immediately

removed from the containers and transferred to a clean dry dessicator for storage purposes. The samples were then analyzed for the hydrogen content one at a time. The analysis time for one complete batch including the NBS sample and any additional required for further verification was approximately one week.

As stated in the proposal, because of the unavailability of the called out grinding wheels the following equivalents were used:

Proposed Wheel	Equivalent Used
32 A46 G10 LV	AA 46 H8V 40
A 46 HV	A 465 J 5 V 30
A 46 MV	DA 60 L 6 V 11

The surface grinder used in this program had a fifty gallon oil sump. However, because of the unavailability of the right quantity of some of the machining fluids, a five gallon tank fitted with a portable pump was used with the surface grinder. The fluid injection rate of the portable pump for a water soluble oil was measured to be 1.82 gallons per minute. (For the same fluid, the injection rate was measured to be 3.33 gallons per minute when the pumping mechanism of the surface grinder was used.) The type and make of the surface grinder and the portable pump are as follows:

Description	Magerle Surface Grinder	Gusher Coolant Pump
Type or Model	FPA 1053	IP3-S (1/10 H.P. motor)
Manufacturer	Magerle Brothers Ltd. Uster, Switzerland	Rulhman Machinery Co. Cincinnati, Ohio

A small vise mounted on the grinding table was used to hold the specimens in place. Prior to mounting and after grinding, the specimens were examined with a micrometer to doublecheck their size and tolerances. Once the specimens were mounted on the vise

great care was exercised to assure that their surfaces were in one plane so that during grinding equal amounts are removed per pass from each specimen. The surface grinder, portable pump, sample mounting configuration, grinding wheel configuration and actual grinding sequences with a directed flow of machining fluid are shown in Figures 4, 5, 6, 7, 8 and 9, respectively.

4.0 EXPERIMENTAL RESULTS AND DISCUSSION

4.1 HYDROGEN ANALYSIS, HARDNESS AND MICROSTRUCTURE OF THE BASE MATERIAL

The hydrogen analysis of the base material from Batch #1 is summarized in Table 4. The mean hydrogen content of six unannealed and six annealed specimens (at 90% confidence level) was found to be 1.49 $\pm$ 0.15 and 0.98 $\pm$ 0.20 ppm, respectively. Base specimens consisting of six unannealed and six annealed samples (Batch #2) prepared from yet another section of the same sheet stock material as Batch #1 gave a hydrogen content reading of 3.06 $\pm$ 0.73 and 3.50 $\pm$ 0.60 ppm, respectively, as shown in Table 5. The high random variation from the mean at 90% confidence level in the above base material should not be considered as unusual. Also the wide variation in the hydrogen readings in the two batches for the respective unannealed or annealed condition is not surprising as in our own laboratories we have frequently observed such variations in the as received material where no specific attempt was made to homogenize the hydrogen distribution.

After the heat treat operation the mean hardness and the corresponding tensile strength of the milled surfaces for the unannealed and annealed specimens were as follows:

Specimen I.D.	Hardness	Tensile Strength, Ksi
Unannealed	51.18	266
Annealed	27.65	128

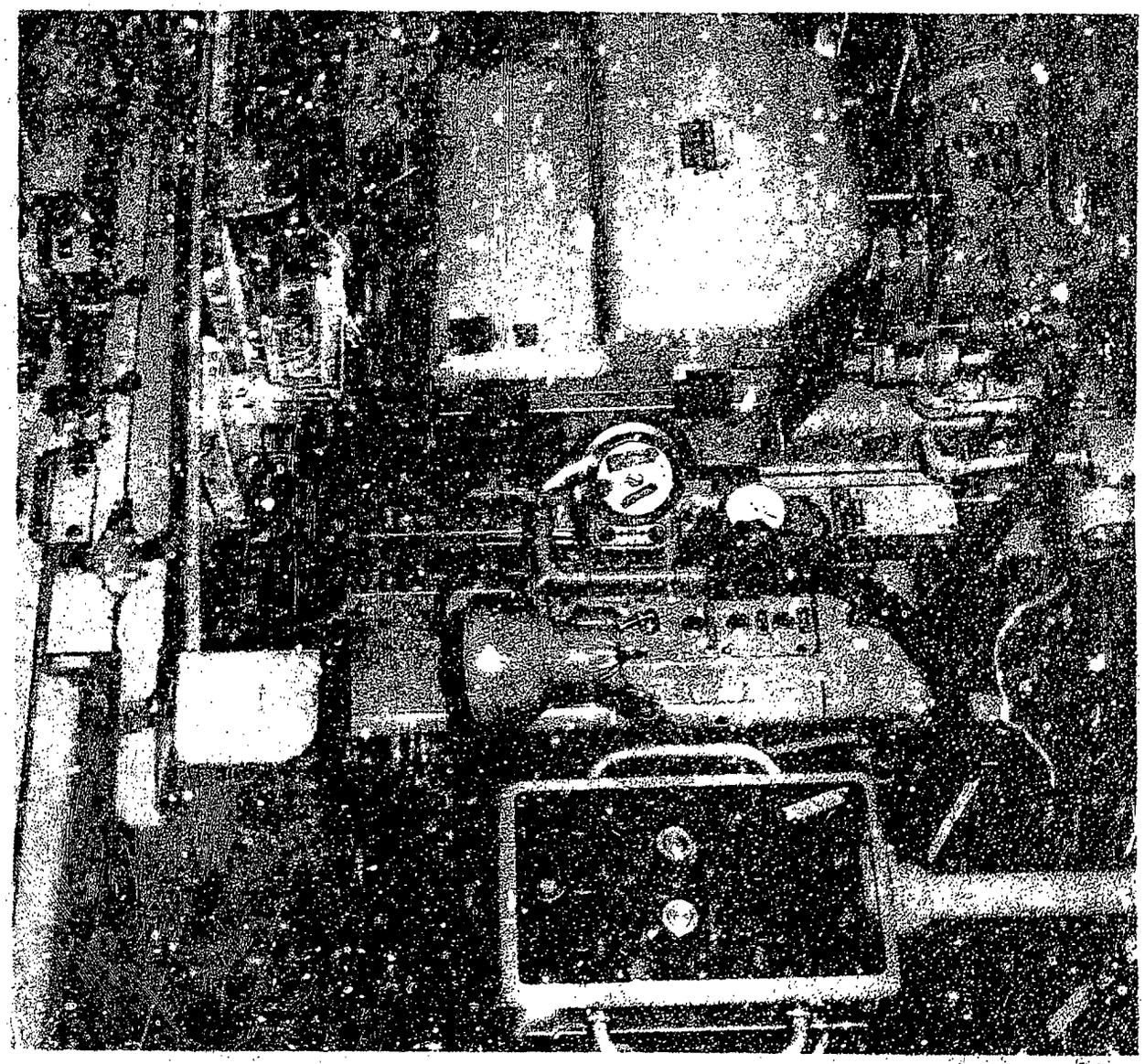

Figure 4: SURFACE GRINDING MACHINE

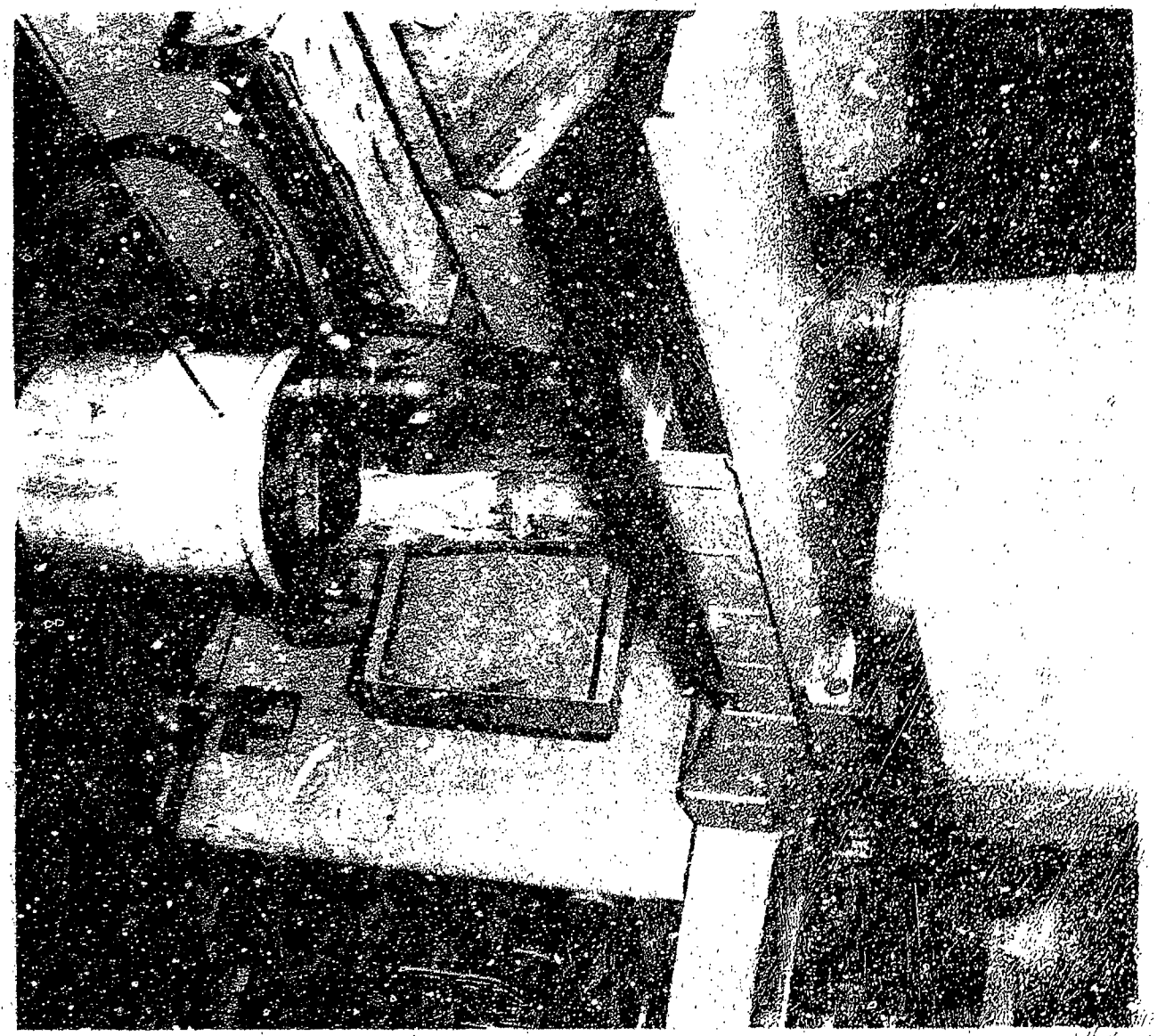

Figure 5 PORTABLE PUMP CONFIGURATION

Figure 6: SAMPLE MOUNTING CONFIGURATION PRIOR TO GRINDING

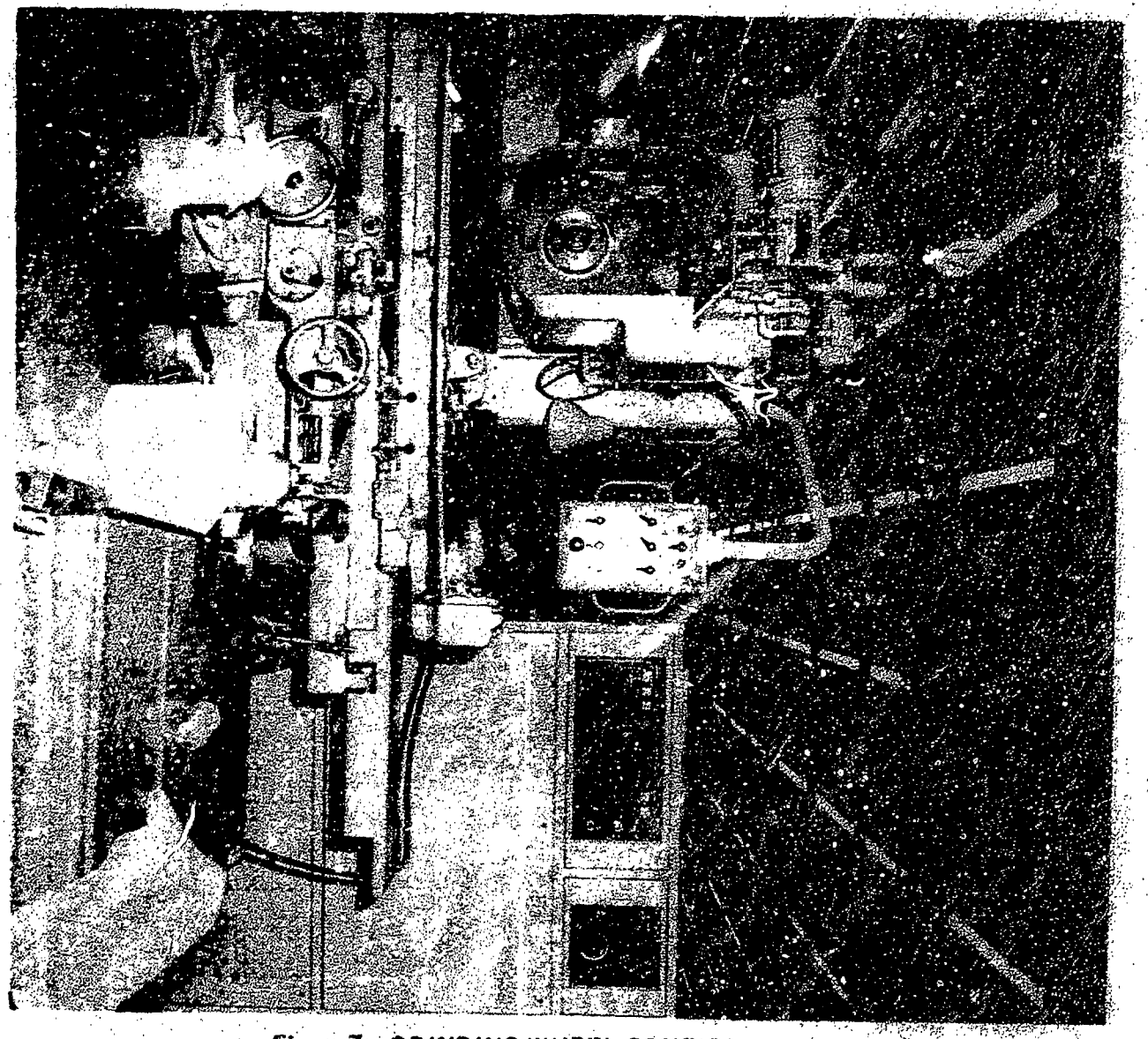

Figure 7: GRINDING WHEEL CONFIGURATION

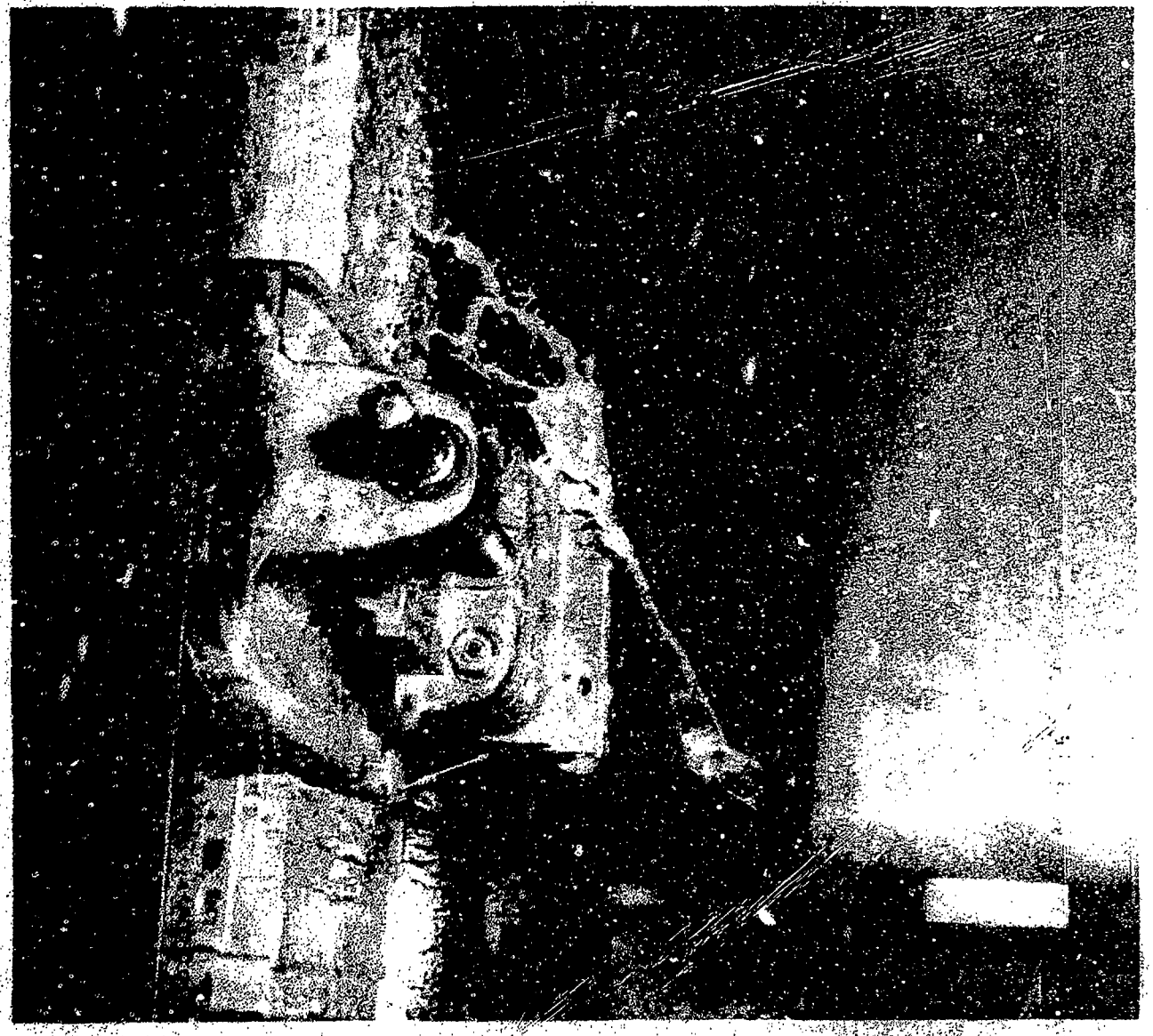

Figure 8: ACTUAL GRINDING SEQUENCE WITH A DIRECTED FLOW OF MACHINING FLUID

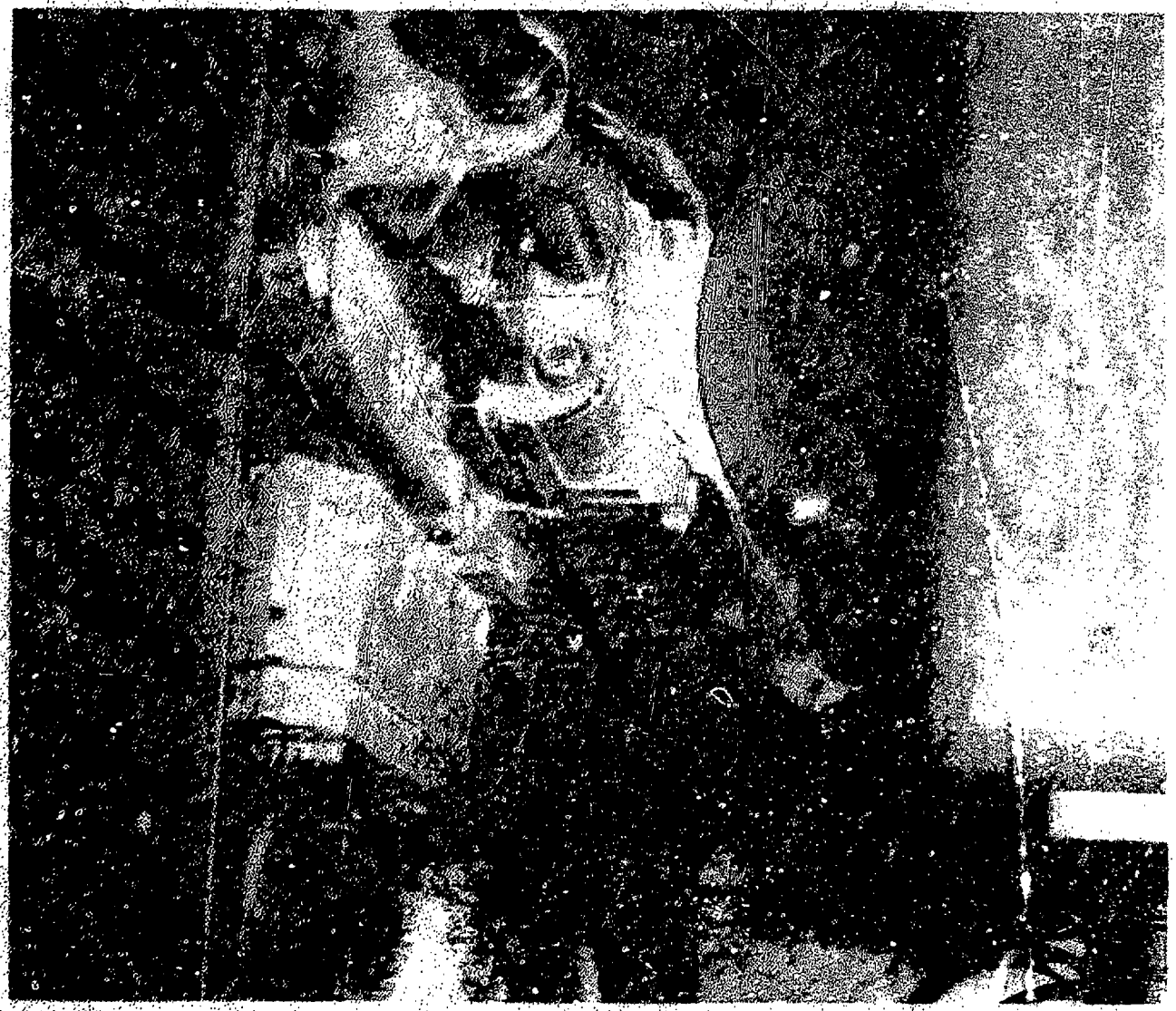

Figure 9: ACTUAL GRINDING SEQUENCE WITH A DIRECTED FLOW OF MACHINING FLUID

The microstructure of these specimens was also examined. Photomicrographs of the structures (at 100X and 500X) for the unannealed and annealed specimens are shown in Figure 10. Both microstructures show fairly comparable grain size. Presence of martensitic structure is also evident in these photomicrographs.

From the data given in Table 5 it can be seen that the hydrogen content determinations of the base material are independent of the two procedures used to clean the specimen surfaces. It should be noted that the cleaning procedure C_2 involves a high temperature operation whereas C_1 cleaning involves the use of degreasing solvents at room temperature.

4.2 HYDROGEN ANALYSIS, HARDNESS AND MICROSTRUCTURE OF MACHINED SPECIMENS

It is known that any process producing atomic hydrogen on the surface of a metal can introduce hydrogen into the metal. Two of the most important functions of a grinding fluid are cooling and lubrication. Cooling protects the abrasive wheel against softening of the bond between abrasive particles, against fracture of the wheel by thermal stresses, and minimized dimensional variation of workpiece caused by thermal effects, thereby stabilizing the depth of cut. Lubrication reduces loading of the wheel face, glazing of abrasive points, and friction between the wheel and the workpiece. However, the heat generated during a typical grinding operation is often sufficient to breakdown the hydrocarbon based grinding fluid thereby providing a source of nascent hydrogen on the grinding wheel-workpiece interface. It has also been reported that residual stresses induced in hardened steel by grinding are influenced by the fluid employed as a result of varying degree of plastic deformation seen by the ground surface. This section describes the results of the experiments in which three different oil base machining fluids and three water-miscible oil emulsion machining fluids were employed to investigate the

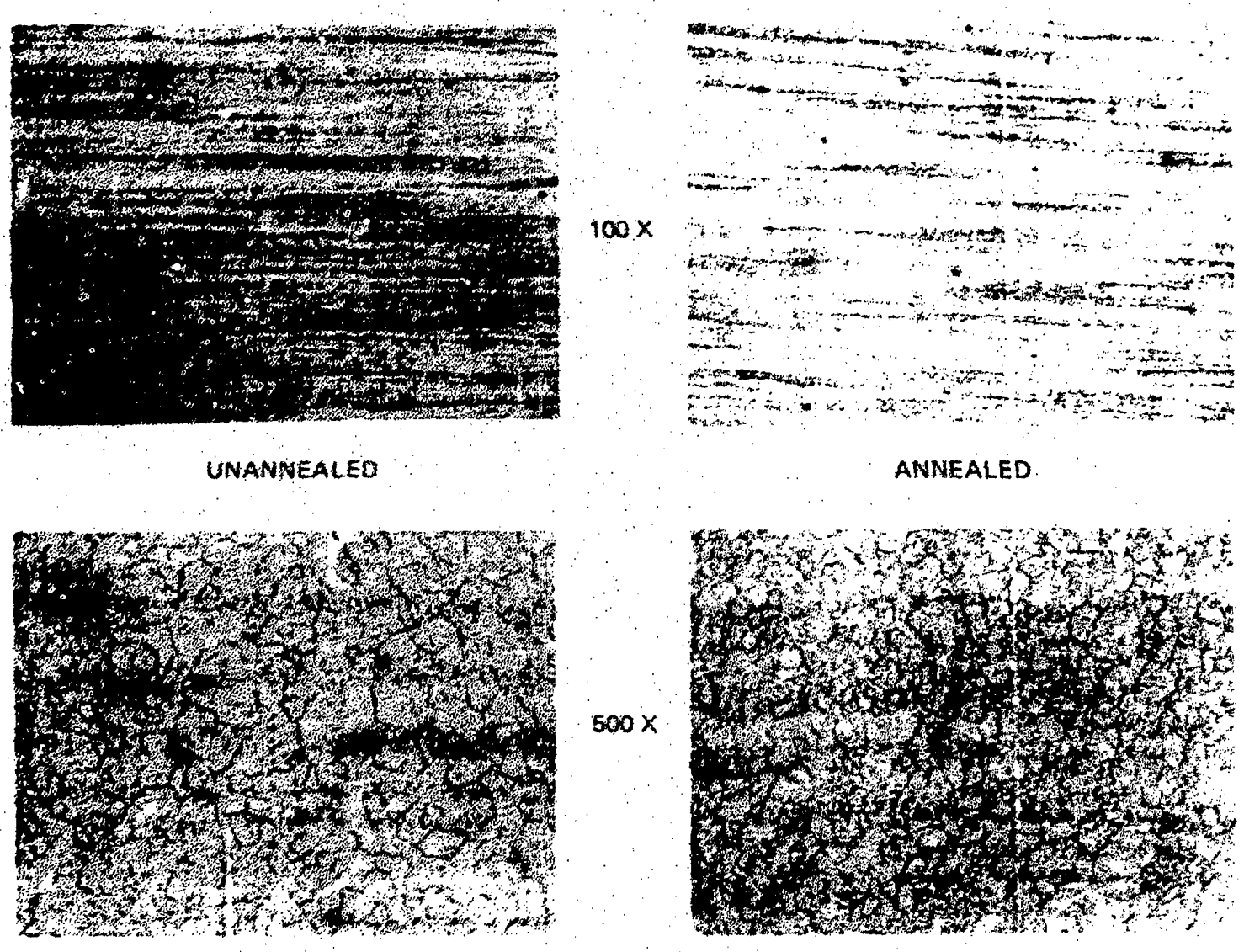

Figure 10: PHOTOMICROGRAPHS OF UNANNEALED AND ANNEALED SPECIMENS

efficacy of grinding fluids on the introduction of hydrogen in 4340 high strength steel during low stress or abusive surface grinding operations. The specimens were prepared and analyzed as per the sequence outlined in sections 3.4 and 3.5.

The mean hardness and the corresponding tensile strength readings of the low stress and abusively ground LSG or AG surfaces of unannealed and annealed specimens are given below:

Specimen I.D.	Hardness, R_c	Tensile Strength, Ksi
Unannealed - LSG	51.7	271
Unannealed - AG	52.4	277
Annealed - LSG	29.0	132
Annealed - AG	29.7	134

The machining fluid in the above case was sulfurized soluble oil. The microstructure of these specimens were also examined. Photomicrographs of the structures (at 100X and 500X) for the unannealed and annealed machined specimens are shown in Figures 11 and 12.

The results of the hydrogen analyses are described in Tables 6 through 29 along with a statistical analysis of the hydrogen data. The tables are arranged in the exact sequence in which the data was obtained. The last column in these tables gives a measure of the excess hydrogen over the base level of the amount of hydrogen pickup by the specimens during machining. The negative readings in this column as seen in Tables 6, 7 and 8 imply that after machining the hydrogen levels in these specimens were lower than the base material. This result was baffling and quite unexpected. Without any further speculation as to what could be happening in the case of chlorinated soluble oil, the system variables were carefully and thorougly checked and its performance was verified by analyzing a number of NBS titanium (hydrogen) standards. The results of the NBS samples were as follows:

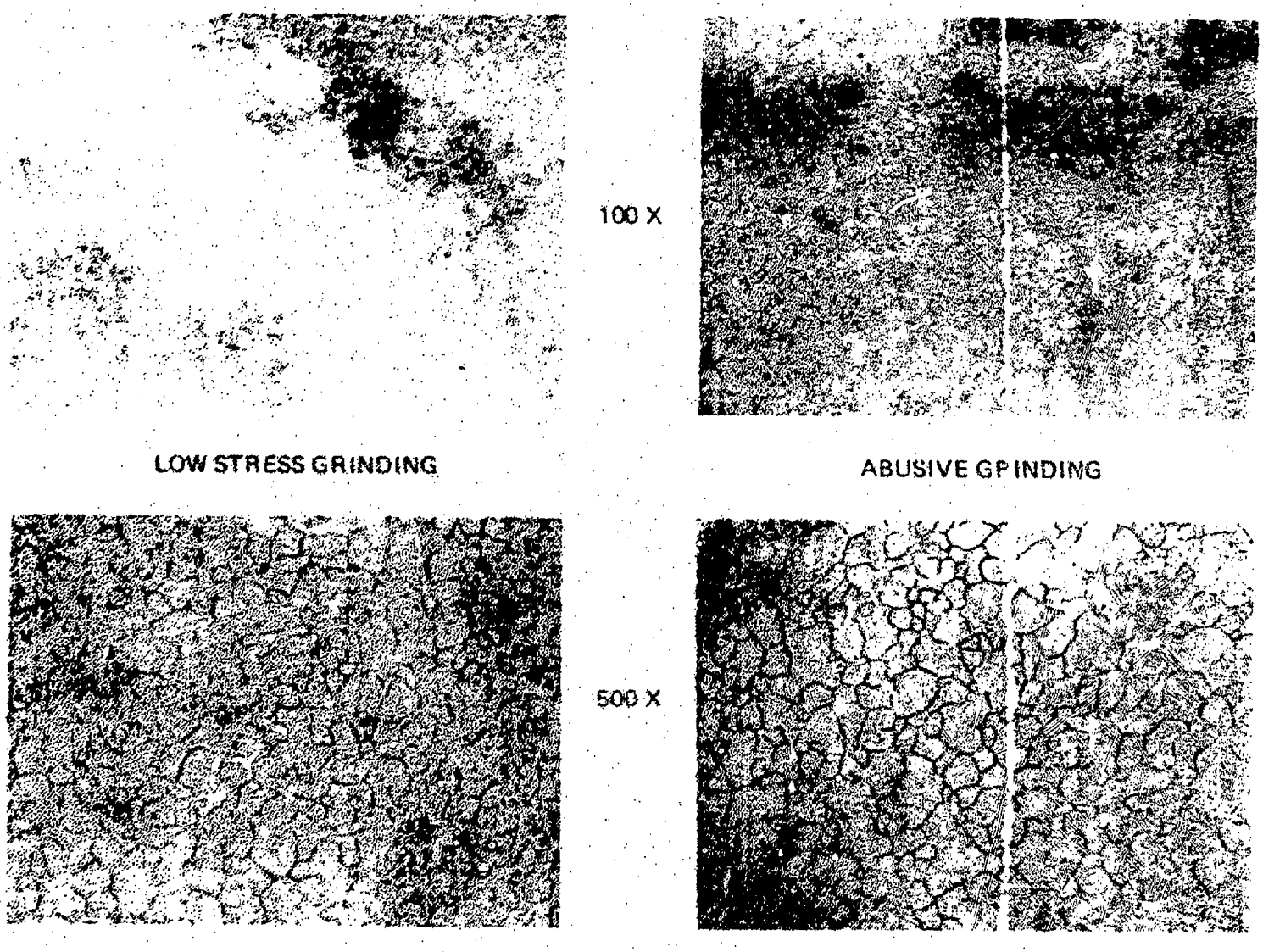

Figure 11: PHOTOMICROGRAPHS OF UNANNEALED SPECIMENS AFTER LOW STRESS AND ABUSIVE GRINDING

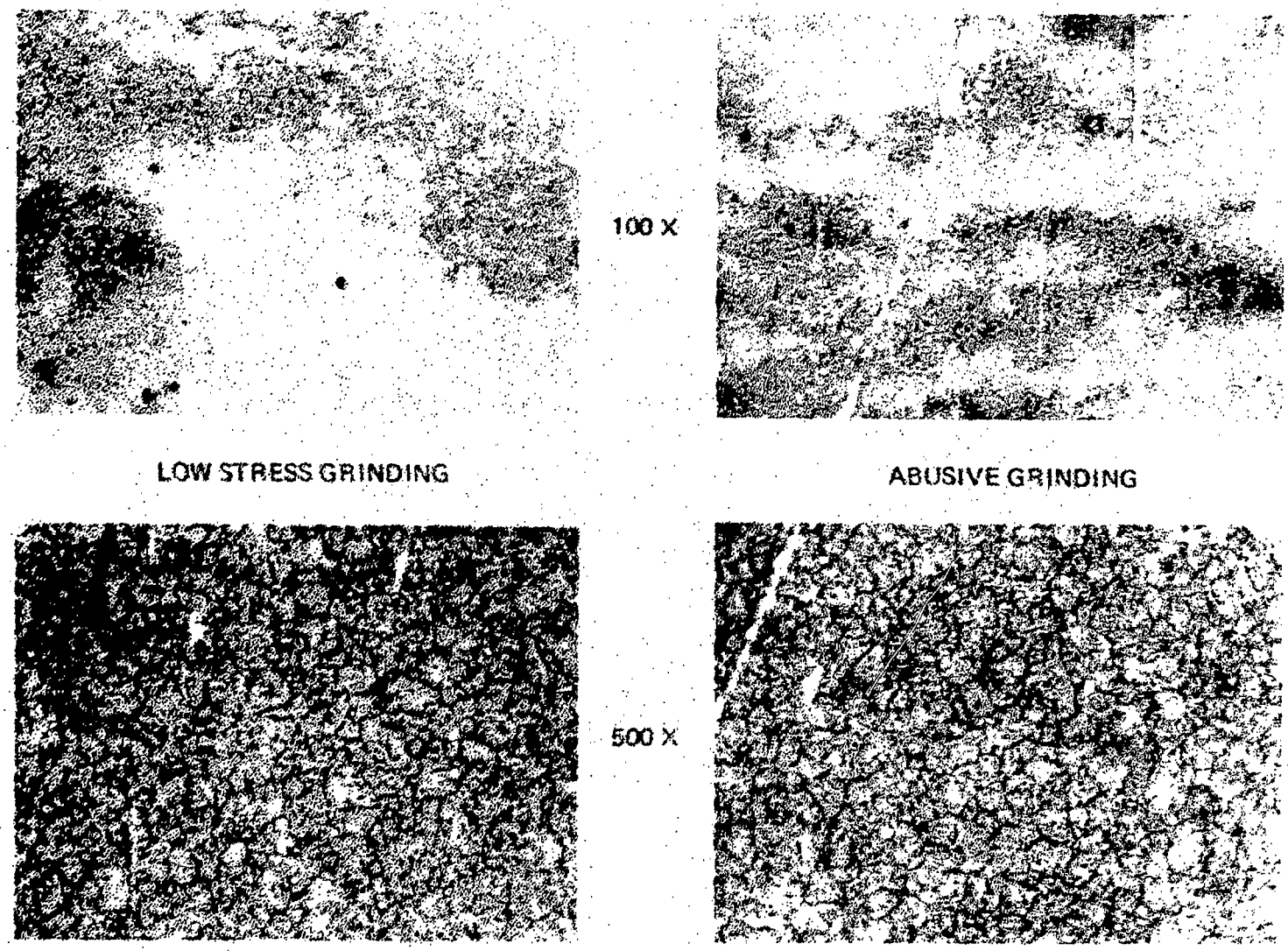

Figure 12: PHOTOMICROGRAPHS OF ANNEALED SPECIMENS AFTER LOW STRESS AND ABUSIVE GRINDING

HYDROGEN ANALYSIS OF THE CERTIFIED NBS SPECIMENS BY "HYDROGEN ANALYSIS SYSTEM"

CERTIFIED NBS VALUE 32 ± 2 PPM	CERTIFIED NBS VALUE 98 ± 5 PPM
33.76	100.42
32.52	108.43
32.73	100.46
34.27	110.99
31.04	95.56
32.84	
36.27	
34.55	

Having checked the system performance in the above manner the rest of the samples machined with the chlorinated soluble oil were analyzed. Furthermore, as a result of such negative readings the total hydrogen value (base hydrogen + hydrogen introduced during machining) was used for the entire set of test data to conduct the statistical analysis so that a meaningful relative comparison of the test results from the different machining fluids could be made.

4.2.1 Chlorinated Soluble Oil (XRJ47C)

The total hydrogen content in this case was observed as follows (see Tables 6 thru 9):

Test Condition	Mean Total Hydrogen Content PPM	Excess Hydrogen Over Base Level PPM
Unannealed - LSG	1.05 ± 0.11	-0.44
Unannealed - AG	1.15 ± 0.15	-0.35
Annealed - LSG	1.08 ± 0.17	+0.10
Annealed - AG	1.25 ± 0.25	+0.27

Here the annealed condition appears to have slightly higher hydrogen content than the base level. However, in the unannealed condition both for LSG and AG operations the readings were lower than the base level. It is interesting to note that not one single reading was above the base level in the unannealed case. The annealed condition gave slightly higher readings but the increase was not significant.

In order to reconfirm the above observations this series of experiments was repeated once more. The results are described below (see Tables 30 through 33):

Test Condition	Mean Total Hydrogen Content PPM	Excess Hydrogen Over Base Level PPM
Unannealed - LSG	4.93 ± 0.76	1.87
Unannealed - AG	5.12 ± 1.36	2.06
Annealed - LSG	3.80 ± 0.74	0.30
Annealed - AG	4.70 ± 0.69	1.20

In this series all the readings in the unannealed condition were higher than the base level but the pattern was not definitive. However, in the case of annealed condition a total of three negative readings were observed. Also, from the above data one would conclude that more hydrogen is picked up by the unannealed specimen than their annealed counterpart because the unannealed specimens have an open structure or a higher density of dislocations than the annealed specimens. It will be seen in the following sections that this observation was not consistent.

In the first set about 58% of the specimens had negative readings whereas in the second set only 8% were below the base level. However, in these runs (Tables 8, 9 and 30 thru 33) there were also quite a few other specimens which had a hydrogen content just barely above the base level. Based on the random variation and the uncertainty limits in the base level data (Tables 4 and 5) it is difficult to attribute such small increases to the

hydrogen pick-up during machining with this soluble oil. Out of the entire set of 48 specimens only 43% were clearly above the uncertainty limits of the base level signifying a positive pick-up of hydrogen.

There were also occasional occurrences of abnormally high hydrogen readings which sometimes approached or went beyond the solubility limit of hydrogen as known for such body centered cubic structures. These readings are marked with asterisks in the Tables. It is interesting to note that there was not one single abnormally high reading in the data described in Tables 6 thru 13. This was first noted when the sulfurized soluble oil series were run and continued to show in the following series including the last run with the chlorinated soluble oil. The origin of this abnormal behavior is not known at this time. Since no such high readings are seen in the base material it appears that this behavior has something to do with the grinding operation. Also the absence of such readings in the first two fluid series could be due to the wheel wear (the experiment was started with brand new wheels). These are only speculations and have to be proven by suitably designed experiments.

In conclusion the following can be said about the specimens ground with the chlorinated soluble oil:

a. Both in the unannealed and annealed cases, specimens subjected to AG operation have a higher total hydrogen content than their respective LSG counterparts.

b. The differences in the total hydrogen content of the unannealed and annealed specimens appear to be random. Thus from this set of data it is difficult to conclude if more hydrogen is introduced in an open structure (unannealed) with a larger dislocation density than in an annealed structure with relatively lower density of dislocations during grinding with chlorinated soluble oil.

c. Since this was the only fluid in the entire series of this program which occasionally gave lower hydrogen readings after grinding than the base level it is speculated that the solution chemistry could be responsible for this behavior. The limited set of data in this program is insufficient to draw any firm conclusion in this regard. In order to explain the above behavior suitable experiments should be designed to answer the following questions:

1. Does this solution act like or have a hydrogen inhibiting agent?
2. Does this solution or some component of it tie up most of the hydrogen introduced during grinding in such a manner as to retain most of it close to the specimen surface? If so, are we losing this excess hydrogen together with some part of the base level during the C_2 cleaning procedure which involves a high temperature cleaning step?
3. Is there a critical ratio of this soluble oil and water in such grinding operations responsible for such abnormal behavior?

d. Finally, if this water soluble machining fluid is indeed instrumental in introducing very little hydrogen or inhibits hydrogen introduction in high strength steels during LSG and AG operations it should be considered as a significant finding.

4.2.2 Neutral Soluble Oil (XRJ47A)

The total hydrogen content in this case was observed as follows (see Tables 10 through 13):

Test Condition	Mean Total Hydrogen Content PPM	Excess Hydrogen Over Base Level PPM
Unannealed - LSG	2.34 ± 0.16	+0.85
Unannealed - AG	2.48 ± 0.30	+0.99
Annealed - LSG	2.71 ± 0.32	+1.22
Annealed - AG	3.32 ± 0.94	+2.34

There were no abnormalities (such as negative or abnormally high readings) observed in this case. Also from the point of view of fractional random variation in the test data this was found to be the best series in the entire program. As can be seen the random variation at 90% confidence level was very close to the variations observed in the base level. On a relative basis both for LSG and AG conditions the hydrogen contents in the unannealed specimens were lower than their annealed counterparts. However, both in the unannealed and annealed cases $(H)_{LSG}$ was less than $(H)_{AG}$.

4.2.3 Sulfurized Soluble Oil (XRJ47B)

Among the soluble oil series this machining fluid showed the highest pick up of hydrogen during the grinding operations. The total hydrogen content in this case was as follows (see Tables 14 through 17):

Test Condition	Mean Total Hydrogen Content PPM	Excess Hydrogen Over Base Level PPM
Unannealed - LSG	5.50 ± 2.29	+4.01
Unannealed - AG	4.27 ± 1.89	+2.78
Annealed - LSG	4.11 ± 1.82	+3.13
Annealed - AG	6.04 ± 2.84	+5.06

Here the fractional random variation was found to be the highest among the soluble oil series. The data clearly shows that among the soluble oils the sulfurized fluid is the one which easily breaks down and introduces significant amount of hydrogen in the test specimens. If the breakdown of water is the chief source of hydrogen in soluble oils then the chemistry of neutral and chlorinated fluids is such that they reduce this breakdown or tie up some of the hydrogen produced during grinding in some chemical manner resulting in lower hydrogen levels in the test specimens. This reaction obviously does not exist in the case of sulfurized soluble oil where high hydrogen levels are seen.

However, on the contrary, if water is not the chief hydrogen producing constituent then the different chemistry of the soluble oils should be responsible for the observed differences.

Another interesting point to note here is the variation in hydrogen levels in the unannealed and annealed specimens. As can be seen above $(H)_{LSGU} > (H)_{AGU}$, $(H)_{LSGU} > (H)_{LSGA}$, $(H)_{AGU} < (H)_{AGA}$ and $(H)_{LSGA} < (H)_{AGA}$. The point of interest is that the same sequence of behavior is observed in the sulfurized oil base series. Also, only two out of 26 specimens in the present case showed abnormally high readings.

4.2.4 Neutral Oil Base

The oil base machining fluid was a mixture of Primol 205 and Metholene 2202. The results of the hydrogen analysis are as follows (see Tables 18 thru 21):

Test Condition	Mean Total Hydrogen Content PPM	Excess Hydrogen Over Base Level PPM
Unannealed - LSG	4.06 ± 0.78	+2.57
Unannealed - AG	5.04 ± 1.74	+3.55
Annealed - LSG	6.59 ± 2.13	+5.61
Annealed - AG	3.20 ± 0.95	+2.31

Only three out of 28 specimens analyzed showed abnormally high readings. Also compared to the neutral soluble oil the random variation from the mean in the present case is considerably higher. As can be seen, both in the unannealed and annealed cases a significant amount of hydrogen is introduced during low stress and abusive grinding operations.

4.2.5 Chlorinated Oil Base

This machining fluid was a mixture of Chlorafin 40, Marcol 52, Marcol 82 and Metholene 2202. The results of the hydrogen analysis are as follows (see Tables 22 through 25):

Test Condition	Mean Total Hydrogen Content PPM	Excess Hydrogen Over Base Level PPM
Unannealed - LSG	4.28 ± 1.39	+2.79
Unannealed - AG	4.87 ± 1.25	+2.38
Annealed - LSG	5.08 ± 1.06	+4.10
Annealed - AG	5.04 ± 1.05	+4.06

Only three out of 27 specimens analyzed showed abnormally high readings. It should be noted that both in the unannealed and annealed cases the hydrogen readings for the LSG and AG conditions are very close and fairly high. Furthermore, it appears that the chemistry of the fluid is such that even in the low stress grinding condition it breaks down quite easily introducing as much hydrogen as in the abusive grinding operation.

4.2.6 Sulfurized Oil Base

In this case the machining fluid was a mixture of Mayco Base 1216, Marcol 52 and Marcol 82. The results of the hydrogen analysis are summarized below (see Tables 26 through 29):

Test Condition	Mean Total Hydrogen Content PPM	Excess Hydrogen Over Base Level PPM
Unannealed - LSG	5.35 ± 1.79	+3.86
Unannealed - AG	5.15 ± 1.31	+3.66
Annealed - LSG	4.99 ± 0.62	+4.01
Annealed - AG	6.00 ± 1.39	+5.02

Only 4 out of 29 specimens analyzed here showed abnormally high readings. Significant amount of hydrogen is introduced in the unannealed and annealed specimens in LSG and AG operations. It may be coincidential but as said previously the trend of hydrogen pickup here is remarkably similar to that seen in the

sulfurized soluble oil case. Also as in the case of sulfurized soluble oil the random variation from the mean in the present case is quite high. Looking at the hydrogen levels it appears that this fluid is also capable of breaking down quite easily in the LSG and AG operations.

4.3 COMPARISON OF THE RESULTS OF WATER SOLUBLE OIL AND OIL BASE MACHINING FLUIDS

Among the water soluble oils all the three fluids behaved differently in as far as the hydrogen levels are concerned. The chlorinated soluble oil showed signs of inhibiting the hydrogen introduction and in cases where there was a positive contribution the hydrogen levels were low. The neutral soluble oil did introduce hydrogen but the range was between 1 to 2 ppm. The sulfurized soluble oil was by far the easiest to breakdown during grinding operations and here the hydrogen levels introduced were between 3 to 5 ppm.

Among the oil base fluids all the three fluids behaved in an identical manner in as far as the hydrogen levels are concerned. All the three fluids (neutral, chlorinated and sulfurized) were able to breakdown during the grinding operations introducing hydrogen levels in the range from 2.5 to 5.5 ppm.

From the statistical analysis of the test data we see that at 90% confidence level the maximum amount of hydrogen introduced is between 5 to 6 ppm. Literature shows that the solubility limit of hydrogen in bcc iron is between 10 to 12 ppm. It is known that for a given available charge of hydrogen, more hydrogen will be absorbed in a plastically deformed structure than in its unworked counterpart. However, the variations in the solubility as a function of the degree of plastic deformation is not clearly known. With the exception of a few abnormally high readings we have undoubtedly not reached the solubility limit in the machined specimens. There is no question that some hydrogen was lost during

the time lapse between the grinding and hydrogen analysis but it is doubtful if this could be as high as 50%. During grinding, since hydrogen is introduced from the surface into the bulk of the material, once the surface is saturated with ionic hydrogen or protons further introduction of protons would experience strong electrostatic repulsive forces. More hydrogen can be introduced only when this surface hydrogen content is changed. This can explain the upper limit of 5 to 6 ppm of hydrogen pick-up for the machined specimens. This limit may vary depending on the degree of severity of the grinding operations and the ease of breakdown of the machining fluid used.

4.4 COMPARISON OF THE RESULTS OF UNANNEALED AND ANNEALED SPECIMENS

With two exceptions in almost all other cases and for both water soluble and oil base fluids a larger amount of hydrogen was introduced into the annealed specimens than in their unannealed counterparts.

It is well known that cold deformation gives rise to an increase in the density of lattice imperfections and therefore, less restricting positions are provided for occupancy of hydrogen atoms[6]. Thus, when compared to the unprestrained steel, the hydrogen solubility in the plastically deformed material is increased, the interaction energy becomes less positive, the entropy is increased or becomes more positive and the susceptibility to hydrogen embrittlement is reduced accordingly. Uhlig, et al[7] reported a marked enhancement of the resistance to cracking of intensely cold worked and hydrogen charged specimens. The specimens made of low carbon martensitic alloy containing 10 to 19 percent nickel were stressed parallel to the rolling direction. The rapidly increasing failure times, measured by Bates[8] on USS 18-2-2 austenitic steels with 10 to 20 percent cold reduction produced after a heat treatment of 1800°F is also explained by the same mechanism when discussed in view of a hydrogen induced failure. The initial decrease in fracture time indicated that the minimum degree of cold deformation must be greater than

10 percent, i.e., a minimum degree of lattice compression is required to make this mechanism applicable.

Let us look at the unannealed and annealed specimen structures in the present case in light of the above discussion. These specimens were prepared by first cutting appropriate sections from the as-received material, milling them to proper size, heat treating to 260-280 ksi strength level and then annealing 50% of the total number of specimens at 1200°F for 3 hours in a vacuum furnace. During the cutting and milling operations the specimens are subjected to severe cold work. This cold work had undoubtedly increased the density of lattice imperfections (dislocations, vacancies and interstitials) in the material. The subsequent heat treatment operation which involves austenizing at 1550°F for one hour and oil quenching, followed by tempering at 425°F for two hours would change the defect structure in the following manner. During the austenizing treatment the excess vacancies and interstitials (introduced by the cold work) would go to sinks, the density of dislocations would decrease but in the oil quenching treatment a part of the density of vacancies in thermal equilibrium at 1550°F would be quenched into the lattice. Unless the quench rate is very rapid the highly mobile vacancies would continue to interact with sinks like dislocations (which may themselves be mobile) during the actual process of quenching and the structure in the final state is left with a complicated array of dislocation network. The following tempering operation at 425°F for two hours removes the excess quenched-in vacancies and rearranges the dislocation network in a more stable configuration. The density of dislocations at this stage would be much smaller than after the cutting and milling operations. Such a structure is referred here as the unannealed state. When this structure is annealed at 1200°F for 3 hours and furnace cooled the density of dislocations is decreased further. In other-words compared to the annealed the unannealed specimens would

have a more open structure. When these two structures are subjected to the same charge of hydrogen the unannealed structure should be able to accommodate or absorb more hydrogen atoms than the annealed structure. In this program, as seen above, the annealed specimens absorbed more hydrogen than their unannealed counterparts when subjected to identical grinding operations. It is assumed here, of course, that during the grinding operation the machining fluid breaks down in such a manner that it provides both the U and A specimens to the same charge of hydrogen. However, surface hardness measurements showed that U were harder than A specimens. Thus, during the grinding operation one would expect the wheel to see a higher resistance to grinding in the U case than in A and as a result more fluid breakdown (higher hydrogen charge) would occur in the grinding of U specimens. However, this argument does not support the present observations. From the observed hydrogen data it appears that during the grinding operations many more dislocations are introduced in A specimens than U resulting in higher hydrogen content in A specimens. In Uhlig's[7] and Bates[8] experiments the specimens were first intensely cold worked and then subjected to hydrogen charge. In the case of U and A specimens the hydrogen charge is taking place concurrently along with the cold work during machining. This could be a very significant difference. Also Bates has reported that the initial decrease in fracture time occurs when the minimum degree of cold deformation is greater than 10 percent. The following questions immediately arise:

1. Instead of annealing the specimens at 1200°F for 3 hours if the U specimens are severly cold worked and then compared with unworked specimens (for hydrogen intake during grinding) would $(H)_{CW}$ be greater than $(H)_{UW}$?

2. Would the enhancement of the resistance to cracking for intensely cold worked and hydrogen charged specimens be different from the case where cold working and hydrogen charging is taking place

concurrently or simultaneously? Would the 10% limit of minimum degree of cold deformation to observe the initial decrease in fracture time as reported by Bates[8] hold good for the case where hydrogen charge is taking place concurrently with cold work?

3. Would the crack propagation rate change as a result of how and when the hydrogen is charged (after coldwork or concurrently with cold work)?

4. Are freshly created dislocations much more active in trapping hydrogen atoms than those which are already present in the specimens?

Answers to these questions can have a significant practical importance in as far as the hydrogen induced damage during fabrication and service conditions of a hardware is concerned.

4.5 HYDROGEN PICK-UP DURING LOW STRESS AND ABUSIVE GRINDING OPERATIONS

The results clearly show that all the machining fluids breakdown during LSG and AG operations introducing hydrogen in the steel specimens. In the majority of cases abusive grinding introduced a higher amount of hydrogen than low stress grinding. This is expected because in abusive grinding a larger amount of hydrogen is generated and the specimen surfaces are subjected to a higher degree of cold work.

For the sake of discussion, let us assume that in the starting material, just prior to grinding, the specimens have a random distribution of hydrogen. During the grinding operation part of the hydrogen generated penetrates the ground specimens. At the end of the grinding operation there may be a concentration gradient of hydrogen from the surface into the bulk of the material. Thus there is a possibility that a small percentage of surface bound or untrapped hydrogen atoms residing in the close proximity of the ground surface may escape during the elapsed time between machining

and hydrogen analysis and during the C_2 cleaning procedure. This could explain the cases where $(H)_{AG}$ was less than $(H)_{LSG}$. It is assumed here that the base level of the specimen in question are the same. It is also assumed that all the specimens subjected to LSG are exposed to an idential charge of hydrogen. The same assumption is made for the AG case.

There is evidence in the literature[1] that specimens containing hydrogen, introduced by any type of charging procedure lose hydrogen when stored at room temperature. The rate of hydrogen loss is determined by a number of variables, particularly composition, temperature, storage time, and to a degree, specimen size. It is understood here that hydrogen that escapes is free to escape. The data in the present case clearly showed that even when the specimens were analyzed after a lapse of two or three days the hydrogen levels were close to that seen on the first day or a few hours after the grinding operation. In fact most of the free hydrogen which could escape would have escaped during the hot alkaline cleaning procedure.

Published literature shows that in the case of high strength steels only 1 to 2 ppm of hydrogen may be detrimental to their strength and could cause hydrogen embrittlement failure. In the present case it is seen that hydrogen levels between 1 to 5 ppm are introduced during the grinding operation. One would tend to believe that in the present case, most if not all the specimens could be susceptible to hydrogen embrittlement failure. Testing for embrittlement failure was of course beyond the scope of this work but some interesting and significant observations were made on the hydrogen evolution rate during the hydrogen analysis which is the subject of discussion of the next section. It should be noted that the hydrogen embrittlement failures referred in the literature are a result of the amount of hydrogen distributed in the bulk or volume of the specimen. During machining, hydrogen is introduced from the surface into the bulk of the material. Thus it is important to know whether most of the hydrogen is residing close to the surface or if there is a concentration gradient from the surface into the bulk of the material. This would have a

bearing on the surface to volume ratio of the charged specimens. For example, in the presence of a gradient, a specimen with a large surface to volume ratio will be more susceptible to hydrogen embrittlement failure than one with a very small surface to volume ratio.

4.6 ESTIMATE OF SURFACE HYDROGEN CONTENT IN MACHINED SPECIMENS

The extremely high sensitivity and the fast detection response of the hydrogen detector requires controlled extraction of hydrogen from the test specimens. Complete extraction is accomplished by totally melting the specimen. Increasing the specimen temperature rapidly to its melting point generally results in the swamping of the detector and in some cases damages the metallic membrane. In cases where the hydrogen content is unknown a calibrated flow division is provided and the temperature of the specimen is raised to its melting point in two or three steps. At low temperatures the surface hydrogen is given off and at high temperatures hydrogen from the total volume or bulk of the specimen is extracted. When the surface hydrogen content is high the temperature is raised in three increments to the melting point of the specimens. For samples with low surface hydrogen the temperature is raised in two steps. Since the hydrogen extraction requires high temperatures the surface hydrogen referred above also contains a certain unknown percentage of the bulk hydrogen.

In the present series of experiments, since the determination of surface contribution was not called for, the specimen ter rature was raised as rapidly as possible (taking care not to swamp the detector) to its melting point. However, a careful examination of the hydrogen analysis curves revealed a very significant phenomenon. With the exception of a few cases in most instances two hydrogen peaks were observed. The first to appear was the low temperature peak where most of the surface hydrogen was given off. Following this, the high temperature peak was observed where the bulk hydrogen was extracted. During surface grinding the breakdown of the machining fluid introduces hydrogen into the specimens. In the present investigation all the machining variables were held the same for different machining fluids used. The purpose of this approach was to measure the differences in hydrogen introduction (total hydrogen

content) as a result of the breakdown of different machining fluids during grinding. A fluid which can easily breakdown is liable to introduce more hydrogen. Larger the breakdown more severe the hydrogen concentration gradient (from the surface into the bulk of the material) is going to be. Furthermore, higher hydrogen concentration on the surface will provide a greater driving force for hydrogen diffusion into the bulk of the material. During low temperature hydrogen extraction a great part of this surface hydrogen is given off but some fraction of this also penetrates into the material. If the specimen is held for prolonged time at this low temperature some bulk hydrogen may also diffuse out. This means that for accurate determination of surface contribution all the specimens should have been taken to the same low temperature and held there for equal lengths of time so that a meaningful relative comparison could be made. However, for the present discussion a very rough estimate of the surface hydrogen content was made for a few machined specimens. Low stress grinding followed by C_2 cleaning procedure was chosen for this discussion. It was also assumed that about 20% of the volume hydrogen content consisted of the surface part which was driven into the material during low temperature extraction. The results of such calculations are given below:

Machining Fluid	Average C_S% $= \frac{\text{Surface H Content}}{\text{Total H Content}} \times 100$	Average Total H Content PPM
<u>Water Soluble</u>		
Neutral	78.2	2.34
Sulfurized	55.8	5.5
Clorinated	38.4	4.93
<u>Oil Base</u>		
Neutral	49.9	4.06
Sulfurized	35.1	5.35
Chlorinated	44.5	4.28

As can be seen, with the exception of neutral soluble oil in all other cases between 35 to 55 percent of the total hydrogen (including the base level) constituted the surface part. In the case of neutral soluble oil about 78 per cent of the total came from the surface. The mere fact that surface hydrogen can be extracted at low temperatures implies that this hydrogen is relatively mobile and free to move around. If such specimens are subjected to stress (soon after machining and cleaning but not subjected to baking) it is this hydrogen which is most likely to migrate to high stress points leading to brittle failure. There is considerable discussion in the literature with regard to the role of locked-in and mobile hydrogen in the embrittlement initiation. The time dependency in sustained load testing and migration of hydrogen to triaxial stress points as proposed by Troiano[9] (where it was shown that crack growth is a discontinuous process) suggests diffusion mechanism of free hydrogen.

The diffusion of free hydrogen to high stress points requires specimen size considerations. In the present case the specimen size was 1/4"x1/4"x1/4" and the specimen weight was about 2 grams. Let us consider, for example, the case of a wedge opening loaded specimen made out of 4340 steel and subjected to identical grinding conditions with the same machining fluids as used in this study. The size of such specimens is normally 3/4" W x 3/4" H x 2" L. The differences in the geometrical size factors are given below:

	Specimen Size (1/4" x 1/4" x 1/4")	Specimen Size (3/4" x 3/4" x 2")
Surface Area of Ground faces (2 faces)	$1/8 in^2$	$3 in^2$
Volume of the Specimen	$1/64 in^3$	$1-(1/8) in^3$
Surface to Volume Ratio	8/in.	2.67/in.
Weight of the Specimen	2 gm	288 gm

For the sake of discussion let us assume that both these specimens have the same level of base hydrogen. Consider the case of the 1/4" cube where the total hydrogen content was 8 ppm (by weight) after grinding

with a given machining fluid. This is equivalent to 16×10^{-6} grams of hydrogen. In other words 128×10^{-6} grams of hydrogen will be introduced for every square inch of surface subjected to the above grinding operation. If the wedge opening loaded specimens are subjected to the same process the amount of hydrogen introduced would be 484×10^{-6} grams or approximately 1.66 ppm (by weight). Now suppose one wants to measure the influence of this excess hydrogen (as introduced during grinding) on the crack propagation rate in the wedge opening loaded notched specimens. If hydrogen is influential in affecting the crack propagation rate, the free or mobile hydrogen will have to migrate to the crack zone when the specimen is loaded. We have seen above that a great percentage of this free hydrogen remains in the close proximity of the surface. Thus if the specimens are tested soon after grinding and cleaning (without baking prior to tests) the free hydrogen will have to migrate a great distance and because of stress distribution may take a very long time to influence the crack rate. Thus not seeing any change during the limited time of testing one may conclude that such a fluid in the grinding operation is not detrimental to the product or there is no concern for the hydrogen induced damage. However, if the same specimen is baked for a short period of time at a low temperature, part of the surface hydrogen will escape and the remaining will diffuse into the material. Testing of such baked specimens is more likely to show changes in the crack propagation rate because of the relatively shorter distances that the hydrogen atoms have to move.

The stress distribution in the wedged specimen is such that the crack zone sees the maximum stress and the outer surfaces of this specimen is subjected to practically minimal stress. As such in the unbaked machined specimens, where most of the hydrogen is in the close vicinity of the outer surfaces, chances are that there is a minimum amount of stress induced migration of this hydrogen from the outer surface into the bulk of the material. However this situation will be totally different in the case of notched tensile specimens. The geometry of the NTS specimens is such that under load the surface and volume both see about the same stress levels. Thus if the NTS specimens are machined

with the fluids used in this program it should be far more easier to check for the hydrogen induced damage or their susceptibility to hydrogen embrittlement failures.

The above discussion is only speculative and should be investigated with suitably designed experiments. Several assumptions were made including the following: (a) same degree of machining is required in the wedge specimens to remove the same amount of material as in 1/4" cubes, (b) the baking temperature and times are such that a part of the hydrogen escapes out and the remaining diffuses into the material to points from where stress induced diffusion to the crack tip is possible, (c) the amount of hydrogen introduced is directly proportional to the area of the machined surfaces and (d) free hydrogen is available which can move under the application of external stress.

Some eight years ago Brittain[10] attempted to correlate the mobile hydrogen content with the delayed failure behavior of a steel alloy. He concluded that it is not the total hydrogen obtained at high extraction temperature which causes hydrogen embrittlement, but rather that portion extracted at a temperature as low as 350°C (the mobile hydrogen). In Brittain's work high strength steel specimens were cyanide cadmium plated, baked, and the delayed brittle failure performance was investigated under sustained loads. After removal of the cadmium plating, the "mobile hydrogen" was determined on the ends of the broken notched tensile specimens. It was assumed here that after plating and baking there was a uniform distribution of hydrogen and a fraction of this hydrogen was mobile or free to move. Apparently under the application of the stress the mobile hydrogen migrates to high stress points resulting in brittle failure. Very low (~.05 ppm) hydrogen contents were reported as being sufficient to induce hydrogen embrittlement.

It is emphasized that the embrittlement determining hydrogen concentration depends on numerous variables such as the composition of steel, its hardness, the hydrogen solubility and diffusivity. Very little is known about the relationship between the hydrogen distribution inside a metal and its embrittlement behavior and the role of total and mobile hydrogen in embrittlement initiation is still open to discussion.

5.0 CONCLUSIONS

The following conclusions are drawn from the test results described in Section 4:

1. Significant amounts of hydrogen was introduced in 4340 high strength steel specimens (heat treated to 260,000 to 280,000 lb/in^2 strength level) during surface grinding operations as a result of the breakdown of the machining fluids. With the exception of a few abnormally high readings, the average upper limit of hydrogen content was 5.5 ppm in the machined specimens. Such high hydrogen levels can cause embrittlement of these specimens.

2. In general it was found that the degree of scatter or the random variation in the test data increased with increasing hydrogen content in the ground specimens. Also from the present series of limited data it is difficult to assess which series of machining fluids (water soluble or oil base) is prone to introducing more hydrogen in ground specimens. This conclusion is based on the following factors:

   a) Unknown percentage of hydrogen lost during the C_2 cleaning procedure which involves a high temperature operation.
   b) Soluble fluids giving high surface hydrogen readings and oil base fluids giving higher total hydrogen readings.
   c) Relatively high randomness or high degree of scatter observed in the data of sulfurized water soluble and all the three oil base fluids.

3. With the exception of a few cases, more hydrogen was introduced during abusive grinding than in the low stress grinding of the specimens.

4. With the exception of a few cases, the hydrogen levels in annealed specimens were either comparable to or slightly higher than the unannealed specimens after machining. This can be due to one or a combination of the following reasons:

a) A 3 hour anneal at 1200°F may not be enough to reduce the dislocation density to a point where significant differences in hydrogen absorbtion during grinding could be seen between the annealed and unannealed specimens.

b) There may be a critical level for the difference in the dislocation density of unannealed and annealed specimens to support the hypothesis that significantly more hydrogen can be absorbed in an open structure in a given surface grinding operation.

c) For a given charge of hydrogen on a specimen surface, more hydrogen may be introduced in the case where hydrogen is charged concurrently with plastic deformation than in the case where the specimens are first cold-worked and then charged with hydrogen.

5. Among the neutral, chlorinated and sulfurized soluble oils, the sulfurized fluid introduced the highest level of hydrogen. Clorinated fluid showed some signs of inhibiting hydrogen introduction.

6. Among the neutral, chlorinated and sulfurized oil base fluids there was not much difference between the hydrogen contents. All the three fluids introduced significant amounts of hydrogen.

7. On a relative basis both in the case of soluble oils and oil base fluids it appears that the sulfurized fluids introduced a high amount of hydrogen.

8. The operating procedure for the hydrogen extraction was such that it was possible to get an estimate of surface or low temperature hydrogen. This was not planned for in this program but for the discussion purposes an estimate of surface hydrogen was made for all the fluids in the case of unannealed specimens subjected to low stress grinding operation. It was found that in the case of neutral soluble oil about 78% of the total hydrogen extracted constituted the surface part. In all other cases the surface contribution was between 35 to 55% of the total extracted.

9. Based on the surface analysis it is speculated that under the application of a stress (for samples with large surface to volume ratio) the relatively mobile surface hydrogen can migrate to high stress points thus initiating the embrittlement of the test specimen.

10. The results of this program have given rise to several questions as described in the text of this report. The answers to these questions can be obtained by suitably designing the test program. As cited by various workers in this field and based on the published data on the subject of hydrogen embrittlement, it is very important that in order to obtain reliable results the test instrument should be capable of measuring low hydrogen contents with high accuracy. The unique features of the present analytical system has not only met this requirement successfully but is capable of providing answers to the questions, which have come up as a result of this test program, in a cost effective manner.

6.0 RECOMMENDATIONS FOR FUTURE STUDY

The results of hydrogen analysis by themselves are of no practical importance unless it is shown that when present in a given amount it can be detrimental to the performance of a product. The critical amount of hydrogen which can embrittle high strength steel structures is not clearly known, although the available data suggest the amount to be in the range of 1 to 2 ppm or less. In the present program it has been shown that as much as 5 to 6 ppm of hydrogen can be introduced in high strength steel specimens as a result of the breakdown of the machining fluids during surface grinding operations. It is also shown that a significant part of this hydrogen resides in the close vicinity of the ground surface. A follow-on to this program should be a study of the notched tensile specimens under sustained loads. The tensile specimens should be prepared by machining them with the same fluids as used in this program. Thereafter, following the conventional sustained load testing procedures, the specimens should be tested for their susceptibility to hydrogen embrittlement failure. The machined specimens should be tested under the following conditions: (a) soon after machining and (b) after machining and baking at a low temperature for a predetermined length of time.

In the machined specimens a hydrogen concentration gradient may exist from the surface into the bulk of the material. Existence of this concentration gradient can be tested by using the laser-microprobe hydrogen analysis system. The procedure will involve carefully sectioning the specimen after machining and then measuring the hydrogen content by scanning the sectioned surface with a .01 in. diameter laser beam using a CW high wattage laser source.

Published literature shows that under the application of an external stress the mobile or free hydrogen moves to the high stress points thus initiating embrittlement in a specimen. This implies that the higher the available mobile hydrogen content, more susceptible the material will be to embrittlement failure. In the present case of machined specimens, large amounts of surface hydrogen was seen to evolve at relatively low temperature which

indicates that under the application of an external stress these specimens can suffer embrittlement failure. Exactly how much of this mobile hydrogen is required to initiate the embrittlement is not known and should be investigated. In general the effect of mobile hydrogen on the mechanical and fracture properties of high strength structural materials requires attention because under the application of an external stress it is this hydrogen which is most likely to cause damage. Some data pertaining to the role of mobile hydrogen can be easily obtained by machining the wedge opening loaded specimens with the machining fluids used in the present program. Subsequent to machining a few specimens should be baked at a low temperature for a short interval of time. The differences in the crack propagation rate in the baked and unbaked wedged specimens should provide some insight into the role of mobile hydrogen. The notched tensile testing experiments as described in the first paragraph of this section should also provide information concerning the mobile hydrogen. It should be noted that under load, the stresses as seen by the surface and internal volume are quite different in the case of a wedged specimen. Whereas, in the case of a loaded NTS specimen the surface and internal volume see almost identical stress levels.

In fact, a slightly modified version of the above experiments should be able to provide extremely useful information with regard to the stress induced and thermally activated diffusivity of hydrogen. For example, suppose a NTS specimen is machined with either water soluble or oil base sulfurized fluid. For the sake of discussion let us assume that all the specimens machined in an identical manner absorb the same amount of hydrogen. When such specimens are loaded for a predetermined length of time, the hydrogen introduced during machining should migrate to the notch area. Thus a measure of the hydrogen content soon after machining and after machining and loading should give an estimate of the stress induced migration of hydrogen into the notch area. Varying the stress level should give a relation between the applied load and the hydrogen concentration in the notch area. In a subsequent experiment similarly machined NTS specimens should be loaded at a given stress level for the same length of time as above. However, in this case under a constant load different specimens should be subjected to different levels of temperature and then analyzed

for hydrogen content. This would give a relationship between the thermally activated hydrogen content that has diffused into the notch zone (under a given constant load) and the operating temperature.

In summary, it is emphasized that the embrittlement determining hydrogen concentration in steel depends on numerous variables. Among them the composition of steel, its hardness, the internal structure (variations in line and point defects), the hydrogen solubility and hydrogen diffusivity are considered to be primary variables. In spite of an enormous amount of research in this area very little is known about the relationship between the distribution of hydrogen inside the metal and its embrittlement behavior. All that is known for certain is that the presence of excessive amounts of hydrogen will make high strength steel structures susceptible to brittle failure. While we are still trying to understand the basic mechanism of hydrogen embrittlement, we should continue to study the behavior of such materials under practical considerations of fabrication and service variables. The experiments described in this section do take into account some of the fabrication and service variables. As a practical application, the results of such studies should contribute directly to the improvement of the integrity and reliability of hardware.

TABLE 1. MACHINING FLUIDS

A.	Oil Base Machining Fluids	Constituent	Trade Name	Producer	Weight
1.	Neutral Oil Base	White Oil	Primol 205	Humble Oil	93.5
		Methyl Lardate	Metholene 2202	Energy Enterprises	6.5
2.	Chlorinated Oil Base	Chlorinated Paraffin Oil	Chlorafin 40	Hercules, Inc.	48.5
		White Oil	Marcol 52	Humble Oil	20
		Methyl Lardate	Metholene 2202	Emery Enterprises	6.5
		White Oil	Marcol 82	Humble Oil	25
3.	Sulfurized Oil Base	Sulfurized Lard Oil	Mayco Base 1216	Mayco Oil & Chemical	40
		White Oil	Marcol 52	Humble Oil	55
		White Oil	Marcol 82	Humble Oil	5

B. Soluble Oil Emulsion Machining Fluids

1. Neutral Soluble Oil Emulsion -
   Base: XRJ47A (Mobil Research & Development Corporation)
   Mixture: One part base to ten parts distilled water

2. Chlorinated Soluble Oil Emulsion -
   Base: XRJ47C (Mobil Research & Development Corporation)
   Mixture: One part base to ten parts distilled water

3. Sulfurized Soluble Oil Emulsions -
   Base: XRJ47B (Mobil Research & Development Corporation)
   Mixture: One part base to ten parts distilled water

TABLE 2. LOW STRESS GRINDING

A. Preliminary Preparations

1. End Milling - to be carried out prior to heat treatment

Cutter diameter, in.	4
Tool material	(370) Carbide
Feed, in/tooth	.005
Cutting speed, ft/min.	200
Tool wear (max.)	.010
Cutter	Six teeth
Fluid	Dry

End milling carried out to reduce specimen thickness to about .030 in. oversize.

2. Grinding - After Heat Treatment

Grinding wheel		(32A46G10LV*)AA46H8V40
Wheel speed, ft/min.		3100 (825RPM)
Depth of grind, in.		.020
	First	.018 in. at .0005 in/pass.
Downfeed, in/pass.	Second	.0008 in. at .0004 in/pass.
	Last	.0012 in. at .0002 in/pass.
Crossfeed, in/pass.		.050
Table speed, ft/min.		40
Fluid		One of prescribed machining fluids in Table 1

Grinding carried out to .010 in. oversize in thickness.

B. Low Stress Grinding Conditions - Final Cuts

Grinding wheel		(A46HV*)A465J5V30
Wheel speed, ft/min.		2000 (540RPM)
Table speed, ft/min.		40
Depth of grind, in.		.010
	First	.0080 in. at .0005 in/pass.
Downfeed	Second	.0008 in. at .0005 in/pass.
	Last	.0012 in. at .0002 in/pass.
Wheel dressing		5 traverses at .001 in. removal per traverse 2 sparkouts
Diamond traverse rate (in/sec.)		1/7
Crossfeed, in/pass.		.050
Fluid		Same as in A.2 (preliminary grinding)

*Not available. An equivalent grinding wheel was used.

TABLE 3. ABUSIVE GRINDING

A. Preliminary Preparations

Same as those outlined in A.1 and A.2 of Table II.

B. Abusive Grinding Conditions - Final Cuts

Grinding wheel	(A46MV*) DA606V11
Wheel speed, ft/min.	6000 (1625 RPM)
Table speed, ft/min.	40
Depth of grind, in.	.010
Downfeed, in/pass.	.002

Wheel Dressing:

5 traverses at .001 in. removal per transverse
2 traverses at .0005 in. removal per traverse
5 traverses at .0002 in. removal per traverse
4 sparkouts

Diamond traverse rate, in/sec.	1/21
Crossfeed, in/pass.	.050
Fluid	Same fluid used in preliminary grinding

*Not available. An equivalent grinding wheel was used.

TABLE 4. HYDROGEN ANALYSIS OF BASE MATERIAL (BATCH #1)

CLEANING PROCEDURE	UNANNEALED SPECIMENS HYDROGEN CONTENT IN PPM	ANNEALED SPECIMENS HYDROGEN CONTENT IN PPM
C_1	1.24	1.08
C_1	1.35	0.84
C_1	1.46	1.04
C_1	1.65	0.79
C_1	1.51	0.73
C_1	1.75	1.39
TOTAL NUMBER OF DETERMINATIONS	6	6
MEAN	1.49	0.98
HIGH VALUE	1.75	1.39
LOW VALUE	1.24	0.84
RANGE	0.51	0.55
STANDARD DEVIATION	0.1879	0.2449
COEFFICIENT OF VARIANCE	12.61%	20.15%
STANDARD ERROR	0.767	0.0999
90% CONFIDENCE LIMITS	$\pm$0.1545	$\pm$0.2015
FRACTIONAL RANDOM ERROR IN MEAN	10.37%	20.56%

TABLE 5. HYDROGEN ANALYSIS OF BASE MATERIAL
(BATCH #2)

CLEANING PROCEDURE	UNANNEALED SPECIMENS HYDROGEN CONTENT IN PPM	ANNEALED SPECIMENS HYDROGEN CONTENT IN PPM
C_1	3.99	2.81
C_1	1.64	4.12
C_1	3.79	4.58
$C_2 + C_1$	2.42	2.83
$C_2 + C_1$	3.23	3.06
$C_2 + C_1$	3.29	3.62
TOTAL NUMBER OF DETERMINATIONS	6	6
MEAN	3.06	3.50
HIGH VALUE	3.99	4.58
LOW VALUE	1.64	2.81
RANGE	2.35	1.77
STANDARD DEVIATION	0.8835	0.7326
COEFFICIENT OF VARIANCE	28.87%	20.93%
STANDARD ERROR	0.3607	0.2991
90% CONFIDENCE LIMITS	$\pm$0.7268	$\pm$0.6027
FRACTIONAL RANDOM ERROR IN MEAN	23.75%	17.22%

Table Number 6 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS

MACHINING FLUID: CHLORINATED SOLUBLE OIL XRJ47C

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1	X								0.82	-0.67
2	X								1.19	-0.30
3	X								1.07	-0.42
4		X							0.99	-0.50
5		X							1.15	-0.34
6		X							1.11	-0.38
7										
8										

TOTAL NUMBER OF DETERMINATIONS η 6

STANDARD DEVIATION σ .1343

COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 12.79 %

STANDARD ERROR $\sigma / \sqrt{\eta}$ .0548

90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{\eta})$ ±.1105

MEAN $\bar{x}$ 1.05 PPM

RANGE 0.37 PPM

HIGH VALUE 1.19 PPM

LOW VALUE 0.82 PPM

FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{x})$ 10.52 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 7: HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: CHLORINATED SOLUBLE OIL XRJ47C

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1			X						0.82	-0.67
2			X						1.09	-0.40
3			X						1.23	-0.26
4				X					1.38	-0.11
5				X					1.17	-0.32
6				X					1.20	-0.29
7										
8										

TOTAL NUMBER OF DETERMINATIONS n 6
STANDARD DEVIATION σ .1869
COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 16.25 %
STANDARD ERROR $\sigma / \sqrt{n}$ .0763
90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{n})$ ± .1537

MEAN $\bar{x}$ 1.15 PPM
RANGE 0.56 PPM
HIGH VALUE 1.38 PPM
LOW VALUE 0.82 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{x})$ 13.37 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 8 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: CHLORINATED SOLUBLE OIL XRJ47C

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1					X				1.12	0.14
2					X				0.85	-0.13
3					X				1.26	0.28
4						X			1.04	0.06
5						X			0.84	0.38
6						X			1.36	-0.14
7										
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ .2128
COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 19.61 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ .0865
90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{\eta})$ ±.1742

MEAN $\bar{x}$ 1.08 PPM
RANGE 0.52 PPM
HIGH VALUE 1.36 PPM
LOW VALUE 0.84 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{x})$ 16.13 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 9 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: CHLORINATED SOLUBLE OIL XRJ47C

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1							X		1.83	0.85
2							X		1.18	0.20
3							X		1.05	0.07
4								X	1.08	0.10
5								X	1.50	0.52
6								X	1.11	0.13
7										
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ .3105
COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 24.07 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ .1268
90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{\eta})$ ±.2554

MEAN $\bar{x}$ 1.29 PPM
RANGE 0.78 PPM
HIGH VALUE 1.83 PPM
LOW VALUE 1.05 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{x})$ 19.80 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 10 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS

MACHINING FLUID: NEUTRAL SOLUBLE OIL XRJ47A

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1	X								2.40	0.91
2	X								2.20	0.71
3	X								2.12	0.63
4		X							2.42	0.93
5		X							2.81	1.32
6		X							2.08	0.59
7										
8										

TOTAL NUMBER OF DETERMINATIONS	η	6	MEAN	$\bar{x}$	2.34 PPM
STANDARD DEVIATION	σ	.2702	RANGE		0.73 PPM
COEFFICIENT OF VARIANCE	$\sigma / \bar{x}$	11.55 %	HIGH VALUE		2.81 PPM
STANDARD ERROR	$\sigma / \sqrt{\eta}$	.1103	LOW VALUE		2.08 PPM
90% CONFIDENCE LIMITS	$(E_c = (t\sigma)/\sqrt{\eta})$	±.1628	FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{x})$		6.96 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 11 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: NEUTRAL SOLUBLE OIL XRJ47A

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1			X						1.88	0.39
2			X						2.44	0.95
3			X						2.20	0.71
4			X						2.47	0.98
5				X					2.97	1.48
6				X					2.34	0.85
7				X					3.04	1.55
8										

TOTAL NUMBER OF DETERMINATIONS η 7
STANDARD DEVIATION σ .4110
COEFFICIENT OF VARIANCE $\sigma / \bar{c}$ 1657 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ .1553
90% CONFIDENCE LIMITS $(E_{\bar{c}} = (t\sigma)/\sqrt{\eta}$ ±.3018

MEAN $\bar{c}$ 2.48 PPM
RANGE 1.16 PPM
HIGH VALUE 3.04 PPM
LOW VALUE 1.88 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{c})$ 12.17 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 12 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: NEUTRAL SOLUBLE OIL XRJ47A

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1					X				2.25	1.27
2					X				3.17	2.19
3					X				2.42	1.44
4						X			2.56	1.58
5						X			3.18	2.20
6						X			2.65	1.67
7										
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ .2885
COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 14.34 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ .1586
90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{\eta}$ ±.3196

MEAN $\bar{x}$ 2.71 PPM
RANGE 0.93 PPM
HIGH VALUE 3.18 PPM
LOW VALUE 2.25 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{x})$ 11.79 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 13 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS

MACHINING FLUID: NEUTRAL SOLUBLE OIL XRJ47A

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1							X		3.33	2.35
2							X		2.29	1.31
3							X		2.60	1.62
4								X	5.45	4.47
5								X	3.51	2.53
6								X	2.76	1.78
7										
8										

TOTAL NUMBER OF DETERMINATIONS η 6

STANDARD DEVIATION σ 1.1370

COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 34.26 %

STANDARD ERROR $\sigma / \sqrt{\eta}$ .4642

90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{\eta})$ ±.9353

MEAN $\bar{x}$ 3.32 PPM

RANGE 3.16 PPM

HIGH VALUE 5.45 PPM

LOW VALUE 2.29 PPM

FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{x})$ 28.17 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 14 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: SULFURIZED SOLUBLE OIL XRJ47B

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1	X								3.48	1.99
2	X								6.23	4.74
3	X								2.86	1.37
4		X							3.21	1.72
5		X							7.44	5.95
6		X							16.29*	--
7		X							9.78	8.29
8										

TOTAL NUMBER OF DETERMINATIONS	η	6	MEAN	$\bar{c}$	5.50 PPM
STANDARD DEVIATION	σ	2.7900	RANGE		6.92 PPM
COEFFICIENT OF VARIANCE	$\sigma / \bar{c}$	50.73 %	HIGH VALUE		9.78 PPM
STANDARD ERROR	$\sigma / \sqrt{\eta}$	1.1390	LOW VALUE		2.86 PPM
90% CONFIDENCE LIMITS	$(E_c = (t\sigma)/\sqrt{\eta}$	±2.2951	FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{c})$		41.73 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 15 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS

MACHINING FLUID: SULFURIZED SOLUBLE OIL XRJ47B

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1			X						2.18	0.69
2			X						2.38	0.69
3			X						8.49	7.00
4				X					4.09	2.60
5				X					3.68	2.19
6				X					4.79	3.30
7										
8										

TOTAL NUMBER OF DETERMINATIONS η 6

STANDARD DEVIATION σ 2.298

COEFFICIENT OF VARIANCE $\sigma / \bar{c}$ 53.82 %

STANDARD ERROR $\sigma / \sqrt{\eta}$ 0.9382

90% CONFIDENCE LIMITS $(E_{\bar{c}} = (t\sigma)/\sqrt{\eta})$ ±1.8904

MEAN $\bar{c}$ 4.27 PPM

RANGE 6.31 PPM

HIGH VALUE 8.49 PPM

LOW VALUE 2.18 PPM

FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{c})$ 44.27 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 16 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: SULFURIZED SOLUBLE OIL XRJ47B

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1					X				4.20	3.22
2					X				2.94	1.96
3					X				7.14	6.16
4						X			2.26	1.28
5						X			6.33	5.35
6						X			1.76	0.78
7										
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ 2.2110
COEFFICIENT OF VARIANCE $\sigma / \bar{c}$ 53.79 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ 0.9026
90% CONFIDENCE LIMITS $(E_{\bar{c}} = (t\sigma)/\sqrt{\eta})$ ±1.8188

MEAN $\bar{c}$ 4.11 PPM
RANGE 5.38 PPM
HIGH VALUE 7.14 PPM
LOW VALUE 1.76 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{c})$ 44.25 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 17 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: SULFURIZED SOLUBLE OIL XRJ47B

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1							X		9.86	8.88
2							X		2.58	1.60
3							X		1.74	0.76
4								X	9.47	8.49
5								X	6.51	5.53
6								X	14.98*	--
7								X	5.98	5.00
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ 3.45
COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 57.43 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ 1.4113
90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{\eta})$ ±2.8438

MEAN $\bar{x}$ 6.02 PPM
RANGE 8.12 PPM
HIGH VALUE 9.86 PPM
LOW VALUE 1.74 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{x})$ 47.24 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 18 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: NEUTRAL OIL BASE

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_R)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1	X								5.18	3.69
2	X								5.23	3.74
3	X								3.61	2.12
4		X							14.43*	--
5		X							3.97	2.48
6		X							3.38	1.89
7		X							2.98	1.49
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ 2.1140
COEFFICIENT OF VARIANCE $\sigma / \bar{c}$ 23.27 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ .3857
90% CONFIDENCE LIMITS $(E_{\bar{c}} = (t\sigma)/\sqrt{\eta})$ ±.7772

MEAN $\bar{c}$ 4.06 PPM
RANGE 6.39 PPM
HIGH VALUE 5.23 PPM
LOW VALUE 2.98 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{c})$ 19.14 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 19 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: NEUTRAL OIL BASE

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1			X						8.53	7.04
2			X						4.41	2.92
3			X						5.34	3.85
4				X					10.52*	--
5				X					4.11	2.62
6				X					5.68	4.19
7				X					2.14	0.65
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ 2.1140
COEFFICIENT OF VARIANCE $\sigma / \bar{c}$ 41.94 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ 0.8630
90% CONFIDENCE LIMITS $(E_{\bar{c}} = (t\sigma)/\sqrt{\eta}$ ±1.7390

MEAN $\bar{c}$ 5.04 PPM
RANGE 6.39 PPM
HIGH VALUE 8.53 PPM
LOW VALUE 2.14 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_{\bar{c}}/\bar{c})$ 34.50 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 20 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: NEUTRAL OIL BASE

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPFCIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1					X				6.69	5.71
2					X				9.51	8.53
3					X				22.20*	--
4					X				5.42	4.44
5						X			5.99	5.01
6						X			2.66	1.68
7						X			9.27	8.29
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ 2.596
COEFFICIENT OF VARIANCE $\sigma / \bar{c}$ 39.39 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ 1.0598
90% CONFIDENCE LIMITS $(E_{\bar{c}} = (t\sigma)/\sqrt{\eta}$ ±2.1355

MEAN $\bar{c}$ 6.59 PPM
RANGE 6.85 PPM
HIGH VALUE 9.51 PPM
LOW VALUE 2.66 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{c})$ 32.41 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 21 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: NEUTRAL OIL BASE

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1							X		2.82	1.84
2							X		2.10	1.12
3							X		2.79	1.81
4							X		5.79	4.81
5								X	2.07	1.09
6								X	3.92	2.94
7								X	3.54	2.56
8										

TOTAL NUMBER OF DETERMINATIONS η 7
STANDARD DEVIATION σ 1.297
COEFFICIENT OF VARIANCE $\sigma / \bar{c}$ 39.42 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ .4902
90% CONFIDENCE LIMITS $(E_{\bar{c}} = (t\sigma)/\sqrt{\eta})$ ± .9524

MEAN $\bar{c}$ 3.29 PPM
RANGE 3.72 PPM
HIGH VALUE 5.79 PPM
LOW VALUE 2.07 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_{\bar{c}}/\bar{c})$ 28.95 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 22 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: CHLORINATED OIL BASE

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1	X								20.20*	--
2	X								2.39	0.90
3	X								3.30	1.81
4	X								6.85	5.36
5		X							5.86	4.37
6		X							3.67	2.18
7		X							3.62	2.13
8										

TOTAL NUMBER OF DETERMINATIONS	η	6	MEAN	$\bar{c}$	4.28 PPM
STANDARD DEVIATION	σ	1.6990	RANGE		4.46 PPM
COEFFICIENT OF VARIANCE	$\sigma / \bar{c}$	39.69 %	HIGH VALUE		6.85 PPM
STANDARD ERROR	$\sigma / \sqrt{\eta}$	.6936	LOW VALUE		2.39 PPM
90% CONFIDENCE LIMITS	$(E_{\bar{c}} = (t\sigma)/\sqrt{\eta}$	± 1.3976	FRACTIONAL RANDOM ERROR IN MEAN	$((E_c/\bar{c})$	32.65 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 23 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS

MACHINING FLUID: CHLORINATED OIL BASE

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1			X						14.03*	--
2			X						3.03	1.54
3			X						6.36	4.87
4			X						6.52	5.03
5				X					3.56	2.07
6				X					5.73	4.24
7				X					4.03	2.54
8										

TOTAL NUMBER OF DETERMINATIONS η 6

STANDARD DEVIATION σ 1.5160

COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 31.13 %

STANDARD ERROR $\sigma / \sqrt{\eta}$ .6189

90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{\eta})$ ± 1.2471

MEAN $\bar{x}$ 4.87 PPM

RANGE 3.33 PPM

HIGH VALUE 6.36 PPM

LOW VALUE 3.03 PPM

FRACTIONAL RANDOM ERROR IN MEAN $(E_c/\bar{x})$ 25.61 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 24: HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: CHLORINATED OIL BASE

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1					X				3.69	2.71
2					X				4.78	3.80
3					X				5.47	4.49
4						X			6.53	5.55
5						X			13.01*	--
6						X			3.59	2.61
7						X			6.44	5.46
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ 1.2860
COEFFICIENT OF VARIANCE $\sigma / \bar{c}$ 25.31 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ 0.5250
90% CONFIDENCE LIMITS $(E_{\bar{c}} = (t\sigma)/\sqrt{\eta})$ ±1.0579

MEAN $\bar{c}$ 5.08 PPM
RANGE 2.94 PPM
HIGH VALUE 6.53 PPM
LOW VALUE 3.59 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{c})$ 20.82 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 25: HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: CHLORINATED OIL BASE

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1							X		3.79	2.81
2							X		5.71	4.73
3							X		2.66	1.6[illegible]
4								X	4.39	3.41
5								X	7.54	6.56
6								X	6.13	5.15
7										
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ 1.763
COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 34.98 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ .7197
90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{\eta}$ ±1.4503

MEAN $\bar{x}$ 5.04 PPM
RANGE 4.38 PPM
HIGH VALUE 7.54 PPM
LOW VALUE 2.66 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{x})$ 28.78 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 26: HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: SULFURIZED OIL BASE

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1	X								6.58	5.09
2	X								5.97	4.48
3	X								9.49*	--
4	X								8.59	7.10
5		X							3.58	2.09
6		X							2.52	1.03
7		X							10.84*	--
8		X							4.85	3.36

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ 2.1810
COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 40.77 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ .8904
90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{\eta}$ ± 1.7941

MEAN $\bar{x}$ 5.35 PPM
RANGE 6.07 PPM
HIGH VALUE 8.59 PPM
LOW VALUE 2.52 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_{\bar{x}}/\bar{x})$ 33.54 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 27: HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: SULFURIZED OIL BASE

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1			X						8.99*	--
2			X						3.25	1.76
3			X						6.96	5.47
4			X						4.57	3.08
5				X					5.66	4.17
6				X					6.83	5.34
7				X					3.62	2.13
8										

TOTAL NUMBER OF DETERMINATIONS	η	6	MEAN	$\bar{\xi}$ 5.15 PPM
STANDARD DEVIATION	σ	1.5910	RANGE	3.71 PPM
COEFFICIENT OF VARIANCE	$\sigma / \bar{\xi}$	30.89 %	HIGH VALUE	6.96 PPM
STANDARD ERROR	$\sigma / \sqrt{\eta}$	.6495	LOW VALUE	3.25 PPM
90% CONFIDENCE LIMITS	$(E_{\bar{\xi}} = (t\sigma)/\sqrt{\eta}$	± 1.3088	FRACTIONAL RANDOM ERROR IN MEAN $((E_{\bar{\xi}}/\bar{\xi})$	25.41 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 28: HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: SULFURIZED OIL BASE

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS)		ABUSIVE GRINDING (UNANNEALED SPECIMENS)		LOW STRESS GRINDING (ANNEALED SPECIMENS)		ABUSIVE GRINDING (ANNEALED SPECIMENS)		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE		CLEANING PROCEDURE			
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1					X				5.38	4.40
2					X				5.34	4.36
3					X				3.76	2.78
4						X			5.76	4.69
5						X			4.39	3.41
6						X			8.84*	--
7						X			5.44	4.46
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ 0.7501
COEFFICIENT OF VARIANCE $\sigma / \bar{c}$ 15.03 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ .3062
90% CONFIDENCE LIMITS $(E_{\bar{c}} = (t\sigma)/\sqrt{\eta}$ ±0.6170

MEAN $\bar{c}$ 4.99 PPM
RANGE 1.91 PPM
HIGH VALUE 5.67 PPM
LOW VALUE 3.76 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{c})$ 12.37 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 29 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: SULFURIZED OIL BASE

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1							X		4.46	3.48
2							X		5.58	4.60
3							X		5.61	4.63
4								X	8.35	7.37
5								X	6.23	5.25
6								X	3.33	2.35
7								X	8.45	7.47
8										

TOTAL NUMBER OF DETERMINATIONS η 7
STANDARD DEVIATION σ 1.8890
COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 31.48 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ 0.7139
90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{\eta})$ ± 1.3871

MEAN $\bar{x}$ 6.00 PPM
RANGE 5.12 PPM
HIGH VALUE 8.45 PPM
LOW VALUE 3.33 PPM
FRACTIONAL RANDOM ERROR IN MEAN $(E_{\bar{x}} / \bar{x})$ 23.12 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 30 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: CHLORINATED SOLUBLE OIL XRJ47C (REPEAT SET)

TRIAL NO.	LOW STRESS GRINDING (INANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1									4.80	1.74
2									5.99	2.93
3									3.31	0.25
4									5.22	2.16
5									5.57	2.51
6									11.94*	-
7									4.74	1.68
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ 0.9268
COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 18.79 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ 0.3784
90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{\eta}$ ±0.7624

MEAN $\bar{x}$ 4.93 PPM
RANGE 2.68 PPM
HIGH VALUE 5.99 PPM
LOW VALUE 3.31 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_{\bar{x}}/\bar{x})$ 15.46 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 31 : HYDROGEN ANALYSIS OF MACHINED SPECIMENS

MACHINING FLUID: CHLORINATED SOLUBLE OIL XRJ47C (REPEAT SET)

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1			X						3.90	0.84
2			X						3.07	0.01
3			X						6.54	2.48
4				X					4.71	1.65
5				X					11.56*	-
6				X					7.51	4.45
7				X					12.65*	-
8				X					5.02	1.96

TOTAL NUMBER OF DETERMINATIONS	η	6		MEAN $\bar{x}$	5.12 PPM
STANDARD DEVIATION	σ	1.6490		RANGE	3.47 PPM
COEFFICIENT OF VARIANCE	$\sigma / \bar{x}$	32.20	%	HIGH VALUE	6.54 PPM
STANDARD ERROR	$\sigma / \sqrt{\eta}$	0.6732		LOW VALUE	3.07 PPM
90% CONFIDENCE LIMITS	$(E_{\bar{x}} = (t\sigma)/\sqrt{\eta}$	± 1.3565		FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{x})$	26.49 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 32: HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: CHLORINATED SOLUBLE OIL XRJ47C (REPEAT SET)

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1					X				2.98	-0.52
2					X				2.55	-0.95
3					X				3.78	0.28
4						X			4.18	0.68
5						X			9.81*	--
6						X			4.94	1.44
7						X			4.40	0.90
8										

TOTAL NUMBER OF DETERMINATIONS n 6
STANDARD DEVIATION σ 0.8989
COEFFICIENT OF VARIANCE $\sigma / \bar{x}$ 23.65 %
STANDARD ERROR $\sigma / \sqrt{n}$ 0.3669
90% CONFIDENCE LIMITS $(E_{\bar{x}} = (t\sigma)/\sqrt{n})$ ±0.7394

MEAN $\bar{x}$ 3.80 PPM
RANGE 2.39 PPM
HIGH VALUE 4.94 PPM
LOW VALUE 2.55 PPM
FRACTIONAL RANDOM ERROR IN MEAN $(E_c/\ )$ 19.46 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

Table Number 33: HYDROGEN ANALYSIS OF MACHINED SPECIMENS
MACHINING FLUID: CHLORINATED SOLUBLE OIL XRJ47C (REPEAT SET)

TRIAL NO.	LOW STRESS GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (UNANNEALED SPECIMENS) CLEANING PROCEDURE		LOW STRESS GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		ABUSIVE GRINDING (ANNEALED SPECIMENS) CLEANING PROCEDURE		HYDROGEN CONTENT IN PPM BY WEIGHT H_M	$(H_M - H_B)$ EXCESS HYDROGEN OVER BASE LEVEL
	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$	C_2	$C_1 + C_2$		
1							X		3.56	0.06
2							X		4.84	1.34
3							X		5.37	1.87
4								X	4.23	0.73
5								X	5.89	2.39
6								X	4.32	0.82
7										
8										

TOTAL NUMBER OF DETERMINATIONS η 6
STANDARD DEVIATION σ 0.8426
COEFFICIENT OF VARIANCE $\sigma / \bar{c}$ 17.93 %
STANDARD ERROR $\sigma / \sqrt{\eta}$ 0.3439
90% CONFIDENCE LIMITS $(E_{\bar{c}} = (t\sigma)/\sqrt{\eta})$ ±0.6931

MEAN $\bar{c}$ 4.70 PPM
RANGE 2.33 PPM
HIGH VALUE 5.89 PPM
LOW VALUE 3.56 PPM
FRACTIONAL RANDOM ERROR IN MEAN $((E_c/\bar{c})$ 14.75 %

*THESE POINTS ARE NOT INCLUDED IN STATISTICAL ANALYSIS

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