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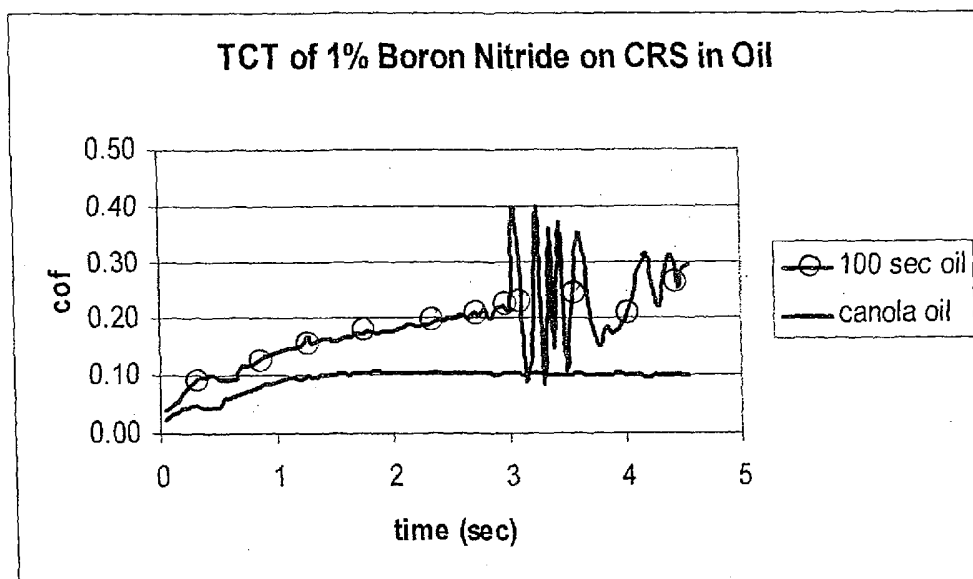
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(54) Title: METAL FORMING LUBRICANT COMPOSITION CONTAINING BORON NITRIDE

TCT Differences As Function of Type of Oil



(57) Abstract: A lubricant for metal forming comprising particles of boron nitride of hexagonal form dispersed in a film of compatible oil of lubricating viscosity is described.



For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

TITLE: METAL FORMING LUBRICANT COMPOSITION CONTAINING
BORON NITRIDE

5

FIELD OF INVENTION

The field of invention is lubricants for metal forming operations such as bending, stamping, and extrusion. The lubricant includes boron nitride in layer lattice or hexagonal form. The lubricant also includes an appropriate liquid carrier in which the boron nitride particles are dispersed.

10

BACKGROUND OF INVENTION

In an article by D.H., Buckley, ASLE Transactions, 21, 1978, pp. 118-124, it maintains that boron nitride (BN) in the layer-lattice or hexagonal form has properties similar to graphite, and consequently should act as a good lubricant. However, lab tests reported in this article indicate that it was not very effective compared to other layer-lattice compounds.

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In J. A. Schey's book, "Tribology In Metalworking," he states on page 49 referring to the article cited above, "The latter two (referring to two fluorinated compounds) have the advantage of being white, as is boron nitride, which had appeared very promising on the basis of its crystal structure yet turned out to be a poor lubricant and now serves as a parting agent that is stable to temperatures over 1000 degrees C."

20

However, more recent work has demonstrated that BN is effective as a lubricant additive. Work reported by Kimura, Y., et al., Wear, 232, 1999, pp. 199-206, indicates that BN has potential as lubricating oil additive.

25

In literature distributed by GE Advanced Ceramics Corporation who markets BN hexagonal powders, BN generated the lowest Coefficient of Friction (cof) in Falex 4-Ball tests compared to other well known solid lubricant powders that included molybdenum disulfide, Teflon (PTFE), antimony oxide, and graphite when compared at 5% levels in an oil.

30

In U.S. patent 5,783,308 entitled "Ceramic Reinforced Fluoropolymer," a combination of BN and particulate fluoropolymer (PTFE) is patented for providing exceptional lubricity properties. This technology is sold by Acheson Colloids Corporation under the trade name Cerflon.

5 However, at this time, there are no known reports of using BN, or Cerflon, in metal forming applications, and there are no reports of any added benefits of using BN as a solid lubricant specifically as an additive in metal forming.

 In difficult metal forming applications, lubricants are used to prevent wear and insure adequate metal deformation. In these applications, significant friction is
10 generated which can cause detrimental wear and heat. Usually a heavy duty lubricant is needed to control the process. These heavy duty forming lubricants are commonly composed of chemistry that contains chlorine, phosphorus, and/or sulfur chemistry. This type of chemistry is referred to as extreme pressure, or just EP, chemistry. Among EP additive chemistry, for example, chlorinated paraffin is
15 known to provide good EP lubrication of over a wide range of operating conditions and temperatures. However, chlorinated and other EP chemistries are perceived by some as an environmental concern, and substitutes are being sought.

SUMMARY OF INVENTION

20 A process for metal forming such as stamping, bending, or extrusion is described wherein the improvement comprises the use of a lubricant that comprises boron nitride particles of the hexagonal or layer lattice type dispersed in a liquid lubricant. A small percentage by weight of said boron nitride dramatically increases the film strength of the lubricant composition during the extreme pressure portions
25 of a metal forming operation. In areas of the metal forming operation not subjected to extreme pressure or heat the liquid lubricant portion of the lubricant composition provides adequate lubrication. The environmentally acceptable lubricant composition replaces current technology which uses chlorinated paraffins and/or a variety of other extreme pressure additives that are not perceived to be as
30 environmentally benign as liquid lubricants and said boron nitride. A preferred liquid lubricant includes polyol esters such as natural vegetable oils. It is also desirable to control the polarity and viscosity of the liquid lubricant such that said

boron nitride remains dispersed in the hydrodynamic lubricating film between the metal being deformed and the equipment applying stress to deform and shape the metal during metal forming.

5

BRIEF DESCRIPTION OF DRAWINGS

Figures 1-7 show the performance (coefficient of friction) of various lubricants in twist compression tests (TCT) under various pressures and conditions. Figure 1 shows a comparison of two different oils with 1% boron nitride. Figure 2 show the effect of pressure with boron nitride. Figure 3 shows the effect of pressure on molybdenum disulfide. Figure 4 shows a comparison of a chlorine containing lubricant to a boron nitride containing lubricant. Figure 5 shows a comparison of a lubricant without boron nitride to one including boron nitride. Figure 6 shows a castor oil based lubricant with different amounts of boron nitride. Figure 7 shows the need to keep the boron nitride dispersed in a lubricating film.

15

DETAILED DESCRIPTION

In the laboratory, experimental lubricants of appropriate polarity and viscosity containing boron nitride (BN) dispersed in liquid lubricant and applied to workpiece metal has been found to respond favorably to increasing pressure, as measured by the twist compression tests (TCT). This combination was found to perform similarly or possibly better than liquid or semisolid lubricants containing extreme pressure (EP) additives without boron nitride. Laboratory twist compression data indicates that the coefficient of friction using BN decreased with increasing pressure to a level that was lower than that generated by a high performance metal forming lubricant containing a significant level of chlorinated paraffin, and also relative to a lubricant generated from a molybdenum disulfide grease. In terms of film strength as measured by twist compression, BN containing lubricants have also responded to pressure in a similar manner to typical EP chemistry.

25
30

Laboratory test results indicate that BN as solid additive to liquid forming lubricants has the possibility for use as a substitute for typical EP chemistry in difficult metal forming applications, and consequently may be used to eliminate the

perceived problems related to environmental, health, disposal issues of chlorinated paraffin and other EP chemistries. BN powders also are preferable to other typical layer lattice additives, such as graphite and molybdenum disulfide in being cleaner and safer to use.

5 Component (a) is an oil of lubricating viscosity. This may be from about 2 to about 2000 cSt at 100 C. These oils may be chosen from any of the American Petroleum Institute base oil categories I-V. Mineral oils and other petroleum distillates function with boron nitride particles of hexagonal form less effectively than oils from polyol esters, thus polyol esters are preferred. The performance of
10 petroleum distillates with boron nitride particles of hexagonal form can be improved somewhat by increasing the viscosity of the oil with viscosity modifiers. The viscosity increase is anticipated to result in better more stable dispersion of the boron nitride through the lubricating film and thereby increases film strength and lubrication performance. Polyol esters of the saturated and unsaturated type are
15 believed to be effective in this application. Saturated polyol esters are believed to have higher thermal stability. Unsaturated polyol esters such as vegetable and animal triglycerides have been found to be particularly effective with boron nitride particles. These may be partially or fully hydrogenated to improve thermal stability and to modify their viscosity. These may be modified with additives known to the
20 art of lubrication. Specific examples of this type of component include oils from canola, castor, soybean or soya, rapeseed, corn, and any other commonly available vegetable oil. Animal triglyceride oils include whale oil, lard, tallow, fish oil, etc.

 The amounts of component (a) in the compositions of the present invention are generally about 1 to 95 or 99.9 % by weight of the lubricious film, more
25 desirably from about 40 to about 80 or 99.9%, and preferably from about 49.5 to about 95 or 99.9 % by weight of the lubricious film.

 Component (b) is particulate boron nitride powder in layer lattice or hexagonal form. Specific examples of this type of component include AC-6003, HCV, and GPC grades available from General Electric Advanced Ceramics. The
30 amounts of component (b) in the compositions of the present invention are generally 0.01 to 10% by weight, desirably about 0.05 to about 2 or 5% by weight, and preferably about 0.1 to about 1, 1.2, or 2 % by weight of the lubricious film. The

boron nitride of hexagonal form can have almost any particle size that can be readily dispersed without undue settling in the oil of lubricating viscosity. Preferred particle sizes for easy dispersion and lack of settling include from about 0.1 to about 100 or 200 microns, more preferably from about 0.1 to about 100 microns in diameter, and most preferably from about 0.1 to about 50 microns in diameter.

Optional component (c) is a polymeric viscosity modifier such as is well known to the lubricant art. These materials help promote the uniform dispersion of the boron nitride in lubricious films such that during extreme pressure situations the boron nitride can promote film integrity. Examples of polymeric viscosity modifiers include polyisobutylene, polymethacrylate, olefin copolymer, esters of styrene-maleic anhydride copolymers, hydrogenated styrene-diene copolymers, hydrogenated radial polyisoprene. Examples of this type of component include polyisobutylene viscosity modifiers having a viscosity of about 2000 cSt at 100 C such as Amoco Indopol H1500.

The amounts of component (c) in the compositions of the present invention are generally about 2 to about 90% by weight, desirably about 2 or 10 to about 60% by weight, and preferably from about 2 or 10 to about 49.5% by weight based on the weight of the lubricious films.

Optional component (d) is a second, third, or more additional oils of lubricating viscosity. These can include petroleum based oils such as mineral oil, and the various higher grade oils of the API base oils. Specific examples of this type of component are typical petroleum base oils, both naphthenic and paraffinic, with viscosities that range from 70 SUS to that of 150 or higher bright stock. The amounts of component (d) in the compositions of the present invention are generally about 1 to about 95% by weight, desirably from about 10 or 20 to about 80% by weight, and preferably from about 10 or 20 to about 60% to about weight based on the weight of the lubricious film.

The particular relationship between the amounts and types of the above components is that the mix of liquid components, such as vegetable, mineral oil, polyisobutylene, etc., need to be of sufficient viscosity to keep the BN uniformly dispersed for application to the workpiece and create a lubricious layer between the BN particles.

The invention is intended for use in metal forming applications such as metal bending, stamping, and extrusion. The invention is also a type of composition that provides enhanced lubricity due to a combination of the quantity, type and properties of hexagonal BN and the lubricating properties provided by the composition of the liquid phase. The BN particles without the liquid phase may not be sufficient at low or high pressures. The liquid phase would have insufficient film strength at higher pressures. The mixed boundary lubrication provided by the liquid phase is as an important component in the reduction of friction as the use of BN powder. The BN powder provides the lubricant film strength that is needed at pressures and temperatures that are similarly provided by EP additives.

Detailed recipes.

Lubricant Composition #1:

15	(a) Canola oil (Cargill Agri-Pure 75)	49.5% by weight
	(b) Polyisobutylene (2000 cSt at 100C)	49.5
	(c) Boron nitride (GE AC6003)	<u>1.0</u>
		100.0

20 Lubricant Composition #2:

	(a) Canola oil (Cargill Agri-Pure 75)	99% by weight
	(c) Boron nitride (GE AC 6003)	<u>1</u>
		100

25 Lubricant Composition #3:

	(d) Rapeseed oil	45% by weight
	(b) Polyisobutylene (2000 cSt at 100C)	45
	(c) Boron nitride (GE HCV)	<u>10</u>
		100

30 Lubricant Composition #4:

	(a) Castor Oil	99% by weight
	(c) Boron nitride (GE HCV)	<u>1</u>
		100

Lubricant Composition #5:

(a) Castor Oil	99.5% by weight
(c) Boron nitride (GE HCV)	<u>0.5</u>
	100.0

5

Lubricant Composition #6:

(d) Petroleum Oil, 2000 sec SUS naphthenic	99.6% by weight
(c) Boron nitride (GE HCV)	<u>0.4</u>
	100.0

10

Lubricant Composition #7:

(d) Petroleum Oil, 100 sec SUS naphthenic	99.6% by weight
(c) Boron nitride (GE HCV)	<u>0.4</u>
	100.0

15

Supplier chemical information

The boron nitride powders used in these studies were provided by GE Advanced Ceramics Corporation. The canola oil was provided by Cargill, and the castor oil by Alnor Oil Company.

20

The tests of the compositions listed on the previous page were conducted using twist compression to measure effects on friction using pressures that are typical of most metal forming operations. The twist compression test (TCT) was generally conducted using a workpiece material of AKDQ cold rolled steel, an annular tool composed of D-2 steel rotated at a rate of 9 rpm and under a pressure of 60,000 psi (60 kpsi or 410 MPa) between the tool and the workpiece, wherein the annular tool has an inner diameter of 19.05 mm (0.75 inch), and an outer diameter of 25.4 mm (1 inch)

25

The overall purpose of the twist compression tests was to demonstrate that hexagonal BN enhances lubricity, in particular that it enhances lubricating properties that effect metal forming. A number of experiments were conducted to demonstrate the properties of BN as a lubricant additive.

30

Experiment 1(Figure 1)

Early work by others showed that BN by itself may not be useful as a lubricant except in extremely high temperature applications. However, BN

dispersed in a fluid has been shown in our work to be a very effective lubricant additive. In order to demonstrate that the nature of the fluid that is used to disperse the BN is important, a comparison of a fluid containing 1% BN in mineral oil (lacking the ability to effectively keep the BN dispersed in the lubricious film) to that of one containing 1% BN in canola oil (**composition #1**) was made using a twist compression test (TCT). Viewing Figure 1, the BN dispersed in canola oil generated a lower cof and greater film strength compared to the same level of BN in 100 SUS naphthenic oil.

Experiment 1 demonstrates the importance of the fluid/lubricant in which the BN is dispersed, and suggests that the better the lubrication that canola oil fluid provides, the greater the combined effect of fluid and BN is to the overall lubrication provided by the dispersed system

Experiment 2 (Figures 2 and 3)

Figure 2 is a set of TCT plots in which **composition #1** was tested at various pressures on AKDQ cold rolled steel (CRS) and tested at a rotation rate of 9 rpm. In these plots, the coefficient of friction (cof) decreases as the pressure was increased. At about a pressure of 157 MPa (22 kpsi), the cof remained approximately constant. The highest pressure used in this series of tests was 258MPa (37kpsi). The shape and the pattern of these TCT plots are typical of compositions using extreme pressure additives, such as chlorinated paraffin, or compositions containing other layer-lattice additives, such as molybdenum disulfide.

(For comparison, Figure 3 is included. It is a set of TCT plots of a forming lubricant containing molybdenum disulfide tested on AKDQ CRS at 9 rpm over a range of pressures. It demonstrates that molybdenum disulfide lubes behaved in a similar manner to the BN test fluid, composition #1.)

Experiment 2 demonstrates that BN dispersed in a relatively good lubricant provides a better combination of mixed boundary and high pressure lubrication, and that these lubricating properties are similar to those provided by extreme pressure additives (EP) and layer lattice lubricant additives.

Experiment 3 (Figure 4)

The EP additive that provides lubrication over the widest range of temperatures and pressures are chlorinated materials. Figure 4 is a comparison of heavy duty forming commercial lubricant that contains 40% chlorine in the form of chlorinated paraffin compared to **composition #1**, a 1% BN dispersion. The 1% BN dispersion achieves a lower cof than the chlorinated fluid indicating its relatively high performance as a lubricant under these test conditions.

Experiment 3 indicates that conditions, that were thought to require chlorinated paraffin, would be adequately meet with a BN dispersion. The use of a BN lube would eliminate the perceived environmental, health and safety issues associated with the chlorinated material.

Experiment 4 (Figures 5, 6)

The level of BN necessary in a lubricant to improve film strength for a specific set of conditions, in this case as a function of pressure and sliding length, was addressed in experiment 4. When viewing TCT data in the form of plots of the change of the cof as a function of time or sliding length, a relatively rapid, often dramatic, increase in the cof is a sign of the failure of the film. (This is often accompanied by acoustic effects and even a change in the speed of the tool rotation.)

Viewing Figure 5, a comparison is made between a liquid vegetable polymer versus the same liquid polymer containing 0.4 by weight BN. This study was conducted at relatively high interfacial pressures, 65 kpsi or 447 MPa. The neat vegetable polymer film broke down rapidly after 60 seconds or after about 9 revolutions of the tool. In comparison, BN helped maintain film strength for a longer period.

In Figure 6, a TCT experiment was conducted after small additions of BN powder to castor oil. This study was conducted at 100 MPa, or 14.5 kpsi, on CRS at 9 rpm. Viewing the plots, the film strength of the straight castor oil and the oil with only 0.007 %, or 70 ppm, by weight BN powder showed failure after about 90 seconds, or 15 revolutions of the tool. The plots of 0.02% and 0.03% by weight of BN showed failure at about 110 seconds, or 16.5 revolutions. The plot of BN containing 0.05% by weight BN showed a relatively slow increase in cof or essentially no film failure for the entire 120 second test. Another factor affected the

plot of the 0.5% BN fluid. If the sample holder is not permitted adequate time to cool down, the test run is conducted at a higher temperature, with reduced fluid viscosity, and generally a correspondingly higher cof is generated as was the case with the run of the 0.5% BN fluid.

5 Experiment 4 demonstrated that BN addition to a liquid lubricant improves the film strength of the lubricant. At lower pressures, such as shown on Figure 6, even small additions in the order of a hundreds of ppm by weight of BN increased the film strength. At higher pressures, both higher levels of BN and better lubrication mixed boundary lubrication are needed to demonstrate improvement in
10 film strength.

Experiment 5 (Figure 7)

This experiment is a TCT plot that demonstrates the change in lubricity in the form of film strength obtained due to the contribution to the mixed boundary lubrication of fluid in which the BN is dispersed. In Figure 7, TCT plots generated
15 by three different fluids are displayed. The low viscosity, poor mixed boundary fluid composed of 0.45% BN in a 100 SUS naphthenic oil failed very quickly. Changing the dispersing fluid to higher viscosity 2000 SUS naphthenic oil, improved the film strength of the lubricant. Changing the dispersing fluid to castor oil and using only 0.03% by weight BN greatly improved the film strength of
20 lubricant, and this fluid ran without film failure for the entire 60 sec test run. (The plot obtained generated by the castor oil plus BN would actually be the same for straight castor oil under these test conditions using TCT.)

The results show that a metal forming lubricant that contains sufficient but not excessive levels of boron nitride powder dispersed in a liquid composition that
25 adequately stabilizes the dispersion will provide lubrication performance properties that are typically associated with lubricants containing common EP additives, and in particular chlorinated paraffin. As EP additives are increasingly associated with environmental problems associated with disposal, the combination of the BN powder being solid, being used at much lower levels, and being relatively
30 environmentally safe recommend its use in heavy duty metal forming applications.

A lubricant of the composition #1 containing 1% by BN described, performs in TCT equivalent to compositions that containing high levels of chlorinated paraffin

or molybdenum disulfide in laboratory twist compression tests. By extrapolation, lubricant composition #1 will permit the elimination of EP chemistry for metal forming lubricants typically required in severe applications.

5 The lubricious films of this disclosure need not have other particulate lubricants such as graphite, molybdenum containing solid lubricants etc. to achieve the necessary lubrication under high pressures. According the limitation consisting essentially of or consisting of may be used in the claims to describe a lubricious film that lacks particulate lubricants other than boron nitride of the hexagonal form. The limitations will not generally exclude common oil soluble additives to lubricants
10 such as antioxidants, dispersants, polymeric viscosity modifiers, detergents etc. The compositions in some embodiments will be free of conventional extreme pressure additives such as sulfur containing extreme pressure additive, chlorine containing extreme pressure additives, and/or phosphorus containing extreme pressure additives. In other embodiments the lubricious films of this disclosure may contain
15 such extreme pressure additives to provide further lubrications effects unique to the combination.

While the invention has been explained in relation to its preferred embodiments, it is to be understood that various modifications thereof will become apparent to those skilled in the art upon reading the specification. Therefore, it is to
20 be understood that the invention disclosed herein is intended to cover such modifications as fall within the scope of the appended claims.

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What is claimed is:

- 5 1. A process for forming or deforming a metal object comprising applying a stress to at least one surface of said metal object forcing metal atoms within said object to move relative to each other to accommodate said stress, wherein a lubricant film is used between said metal object and one or more surfaces that it comes in contact with said metal during said deforming step, wherein the improvement in said process is including within said lubricant film boron nitride particles of the
10 hexagonal form to provide film integrity during said deforming step.
2. A process according to claim 1, where said boron nitride is present from about 0.05 to about 5 weight percent based on the weight of said lubricant film.
- 15 3. A process according to claim 2, wherein said lubricant film further comprises from about 2 to about 60 weight percent of a polymeric viscosity modifier.
4. A process according to claim 2, wherein said lubricant film comprises
20 at least 40 weight percent oil of lubricating viscosity.
5. A process according to claim 4, wherein said oil of lubricating viscosity comprises at least 40 weight percent of a polyol ester lubricant.
- 25 6. A process according to claim 5, wherein said polyol ester lubricant comprises a vegetable or animal triglyceride oil, optionally partially or fully hydrogenated.
7. A process according to claim 2, wherein said lubricant consists
30 essentially of one or more oils of lubricating viscosity, 0.01 to about 2 weight percent of boron nitride particles of hexagonal form, and optional oil soluble

additive(s) selected from the group consisting of dispersants, antioxidants, detergents, friction modifiers, extreme pressure additives, and viscosity modifiers.

5 8. A process according to claim 2, wherein at least a portion of said lubricant film is subjected to a compressive force between said metal object and said one or more surfaces that it comes in contact with of at least 17 Ksi.

 9. A process according to claim 8, wherein said portion of said lubricant film under said pressure of at least 17 Ksi is also subject to shear forces
10 simultaneously with said compressive force.

 10. A process according to claim 8, wherein said hexagonal boron nitride incorporated into a forming lubricant is associated with an increase in the lubricant film strength as characterized by at least a 50 % increase in time to film failure in a
15 twist compression test (TCT) using a workpiece material of AKDQ cold rolled steel, an annular tool composed of D-2 steel rotated at a rate of 9 rpm and under a pressure of 60,000 psi (60 kpsi or 410 MPa) between the tool and the workpiece, wherein the annular tool has an inner diameter of 19.05 mm (0.75 inch), and an outer diameter of 25.4 mm (1 inch) as compared between the lubricant with and without the boron
20 nitride component

 11. A lubricant for metal stamping, bending, or extruding comprising
 a) about 0.05 to about 5 weight percent of particles of boron nitride of hexagonal form, and
25 b) an oil of lubricating viscosity forming a continuous phase that has sufficient compatibility with said boron nitride or viscosity to keep said particles of boron nitride operationally dispersed at least 20 seconds in a twist compression test using a cold rolled steel workpiece and an annular tool composed of D-2 steel with an inner diameter of 19.05 mm and an outer diameter of 25.4 mm rotated at a rate of 9 rpm under
30 60,000 psi pressure.

TCT Differences As Function of Type of Oil

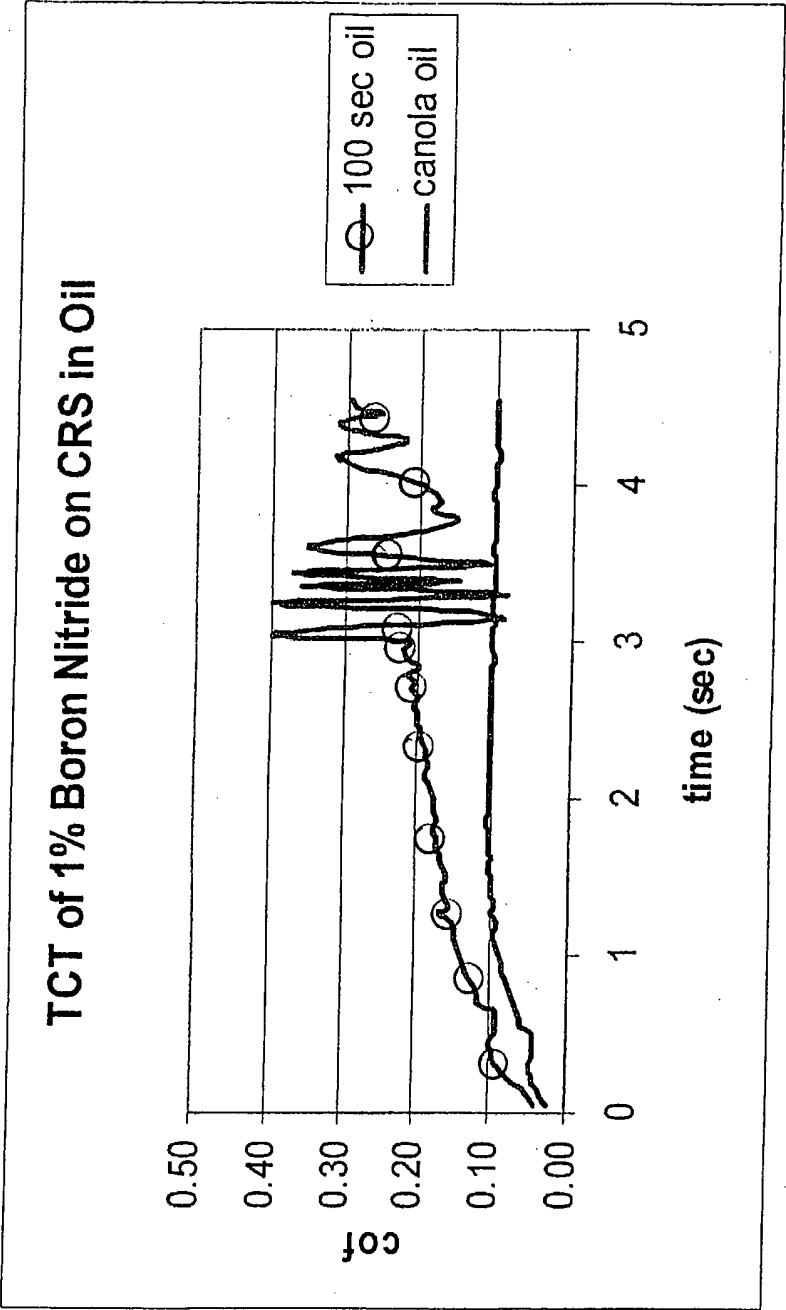


Figure 1

TCT of Boron Nitride Containing Oil As A Function of Pressure

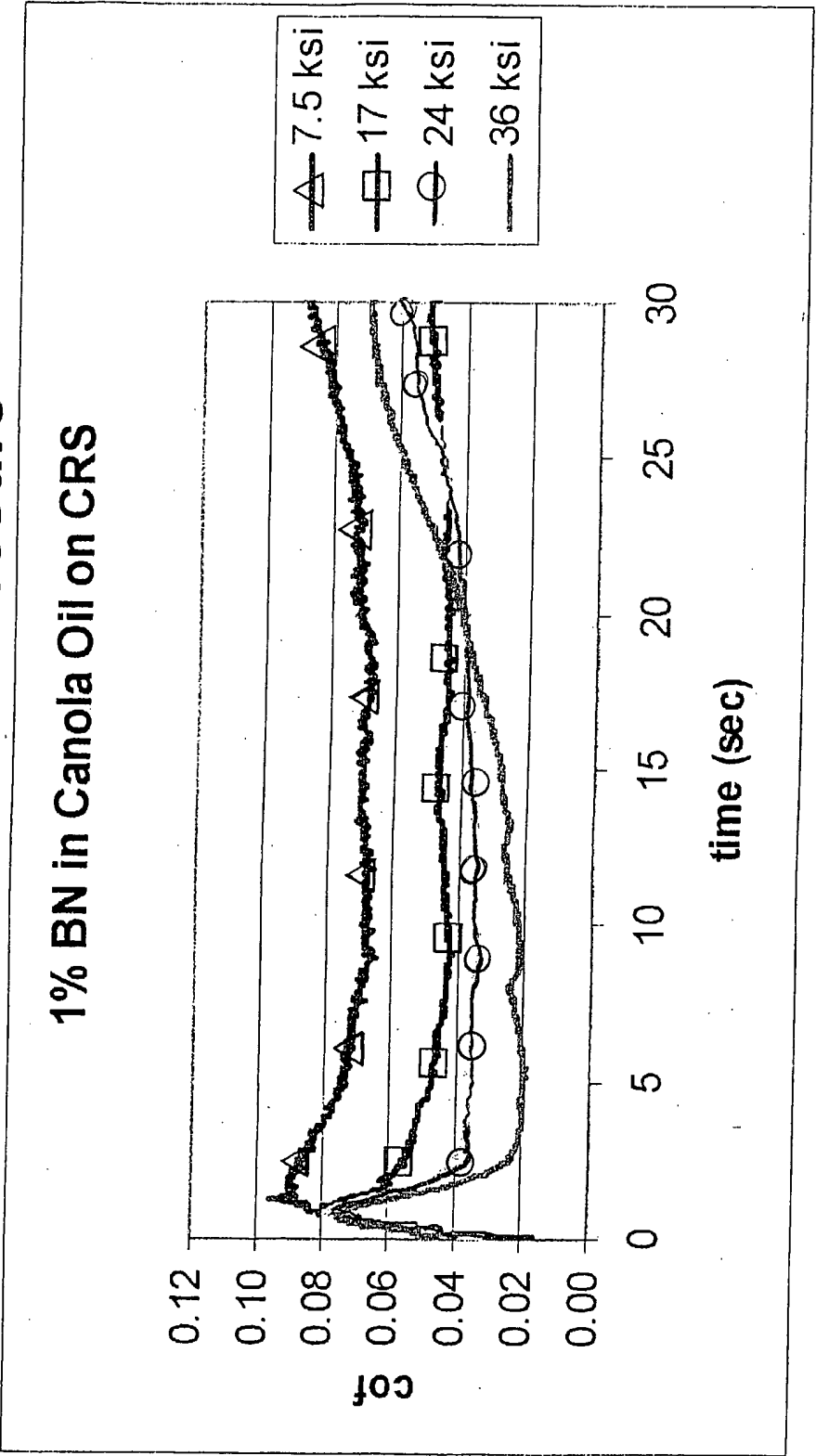


Figure 2

TCT of MoS₂ As A Function of Pressure

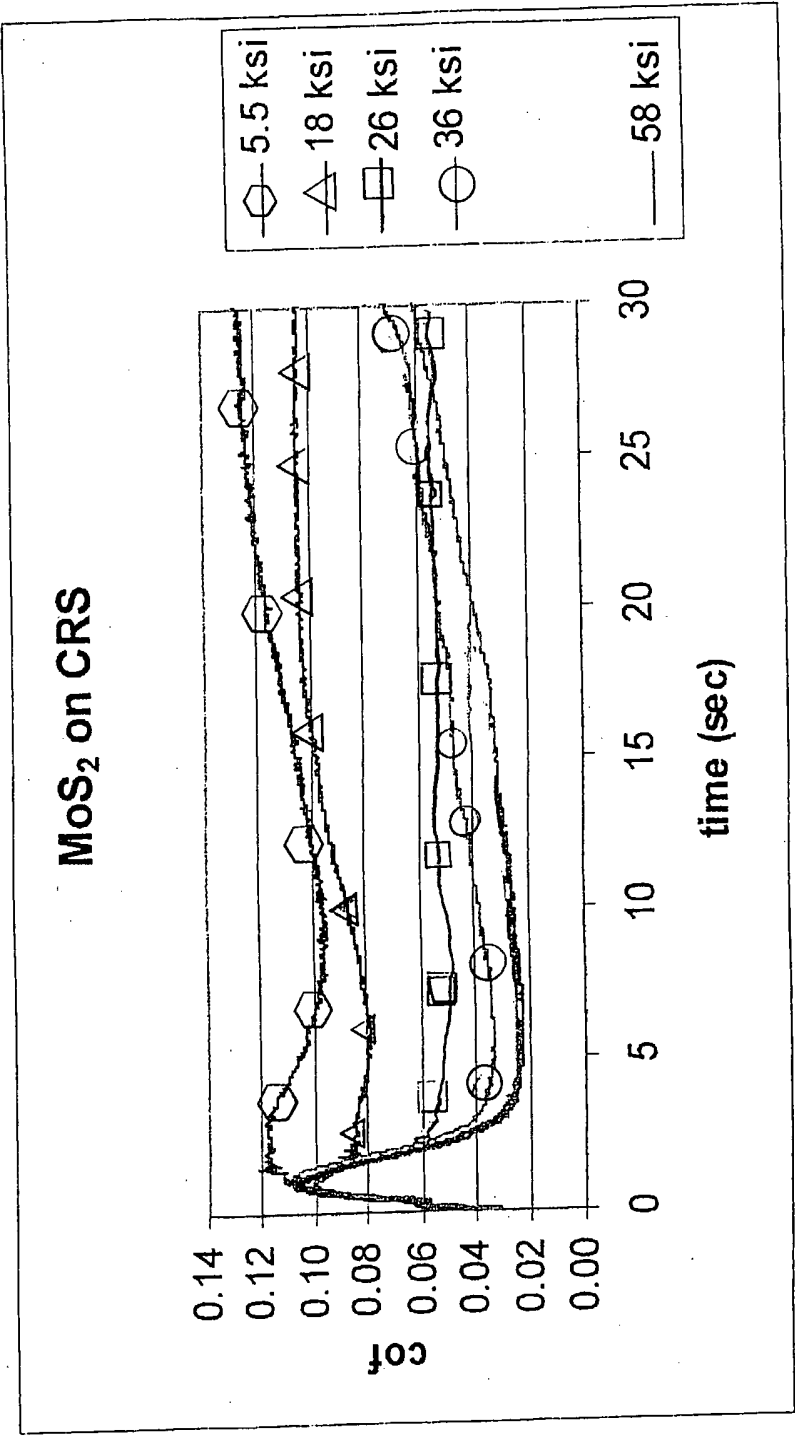


Figure 3

Comparison of TCT on CRS at 30 ksi

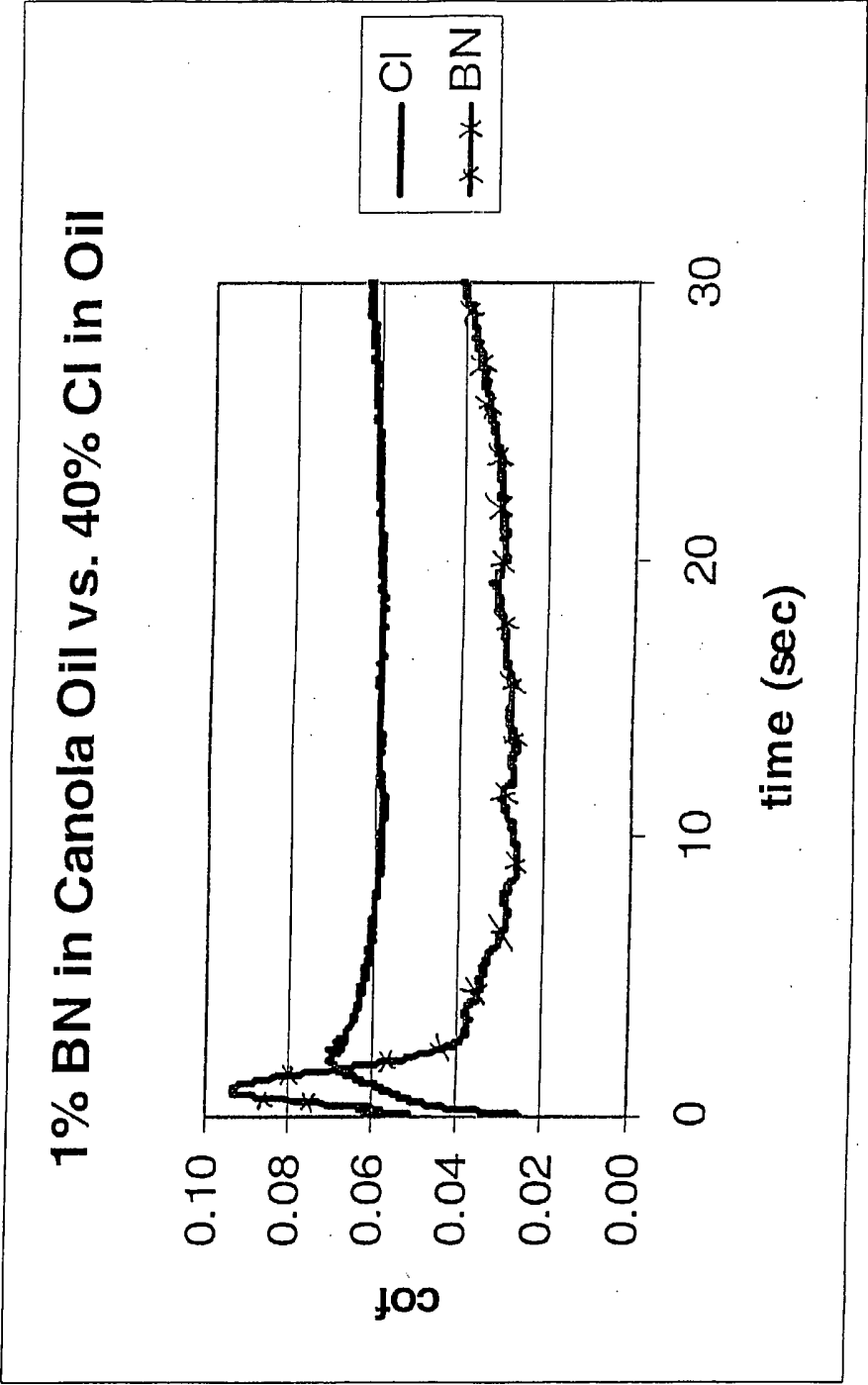


Figure 4

Vegetable Polymer on CRS at 65 ksi (447 MPa)

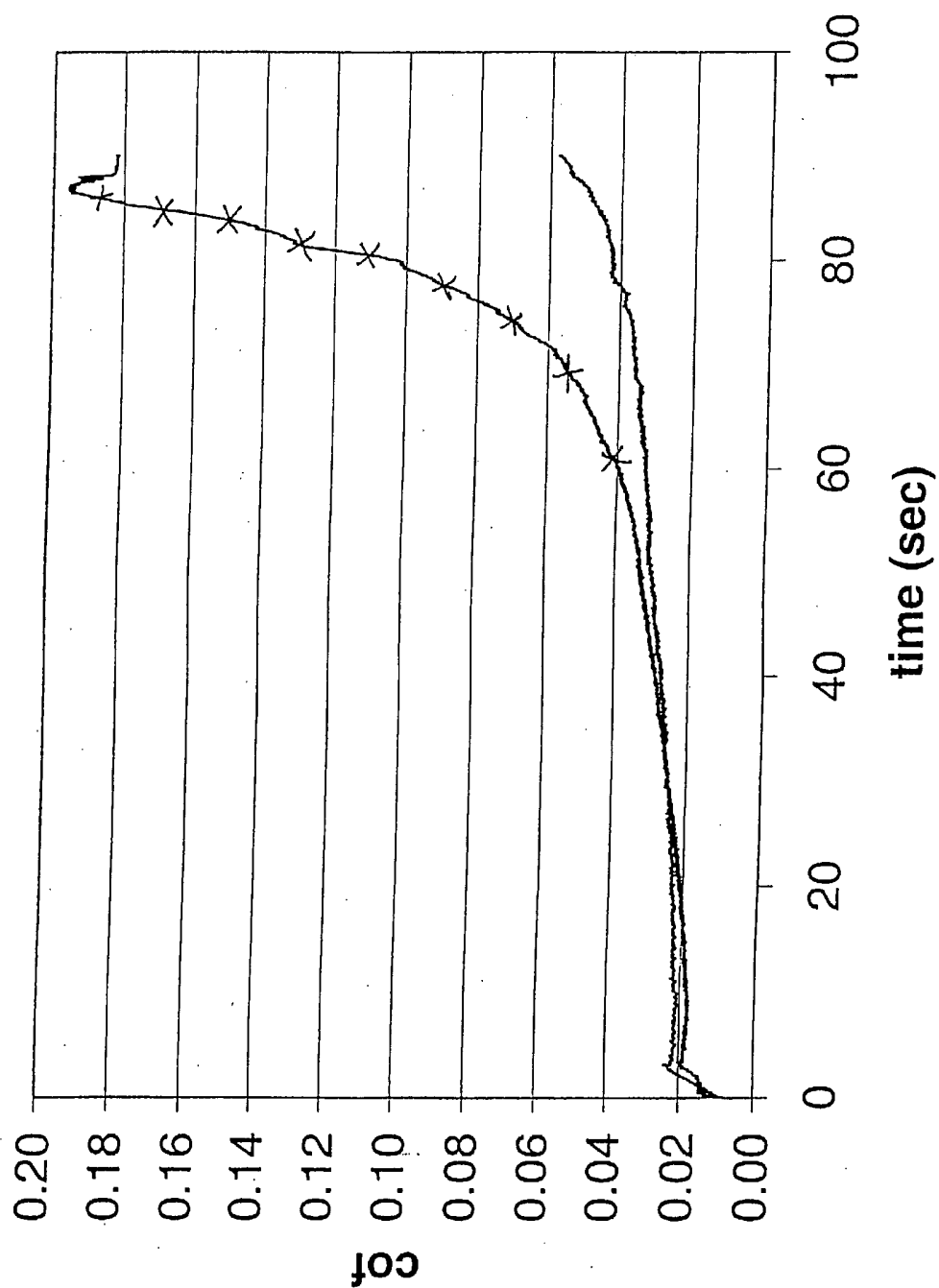


Figure 5

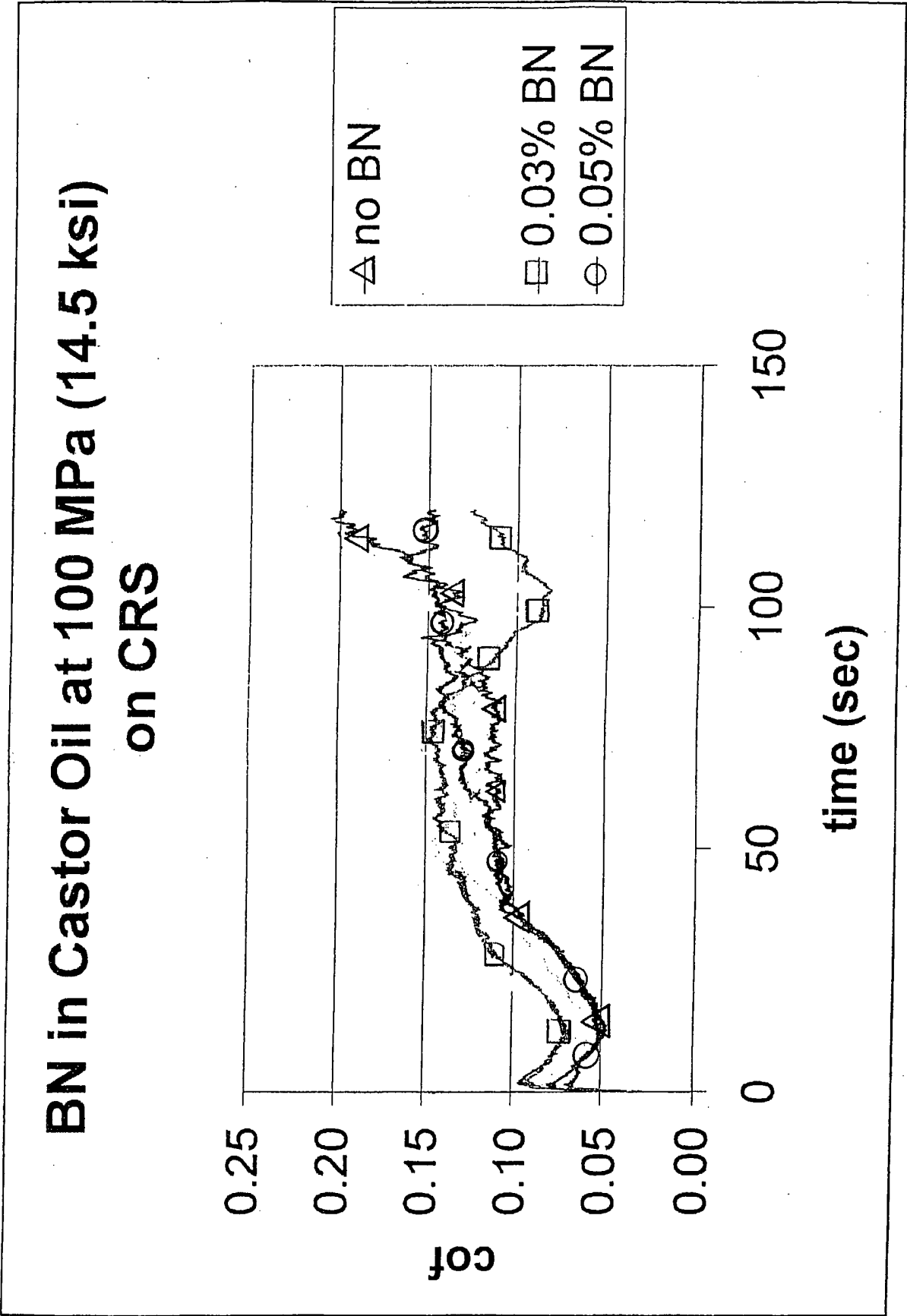


Figure 6

**Effect of Dispersing Fluid
at 20 ksi on CRS**

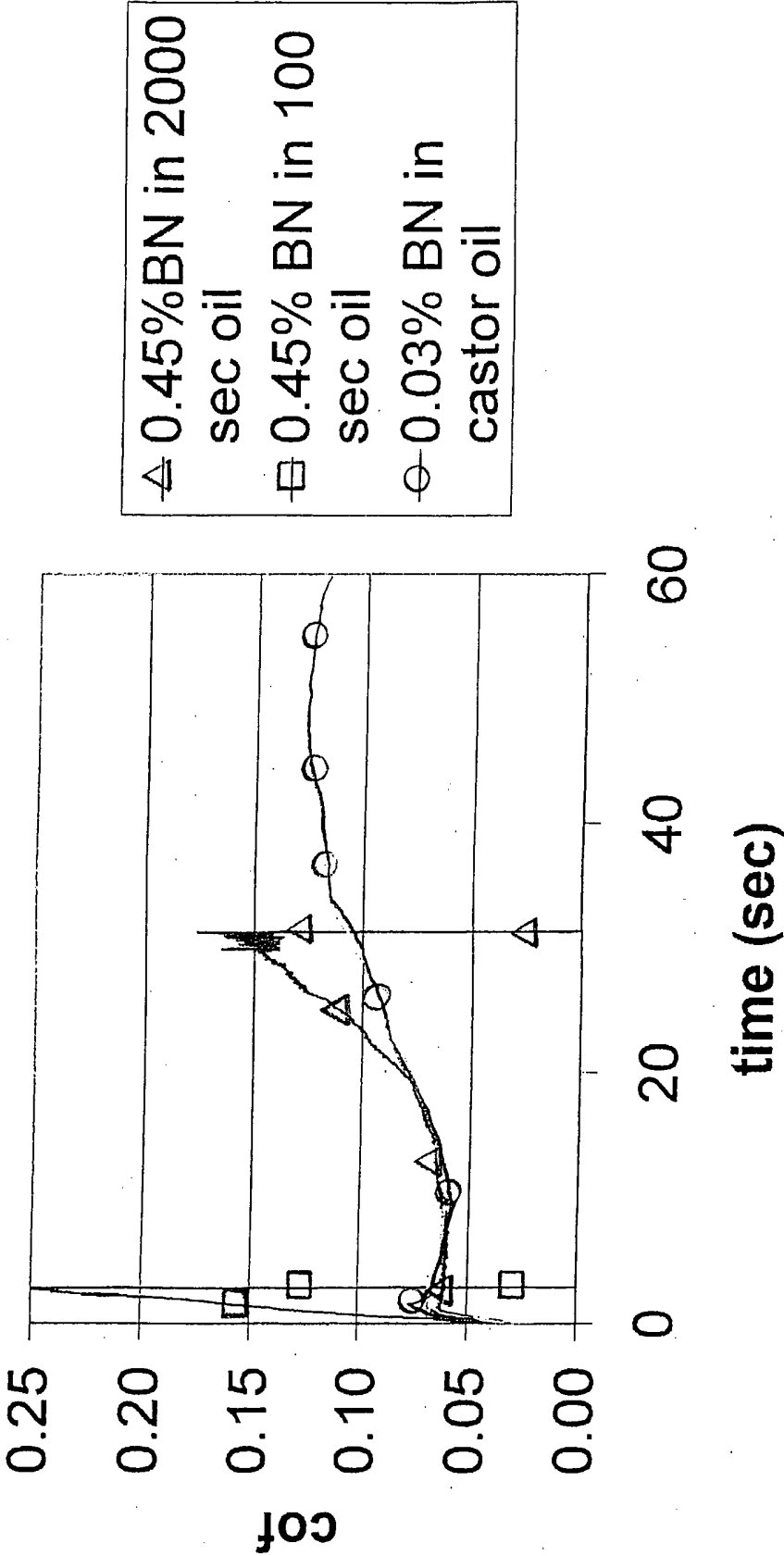


Figure 7

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US05/15128

A. CLASSIFICATION OF SUBJECT MATTER
IPC(7) : C10M 125/26, 105/38; B21B 45/02
US CL : 508/155, 485, 491, 591; 72/42

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EAST search terms: boron nitride, lubricant film, polyol ester, metal (forming or deforming)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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A	US 4,128,486 A (PALMER et al) 05 December 1978 (05.12.1978), column 1, line 5 to column 2, line 53.	1-11

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

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