Evaluation of lubricant viscosity and base oil effects by form tapping test.

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Summary

In order to correlate the effect of lubricant viscosity and base oil on the efficiency of a lubricant, we measure the variation of tapping torque according to ASTM D5619 standard. Three fully formulated lubricants containing the same additive package but having different viscosities are tested in the first part of the experiment. The effect of chemically active and chemically non-active paraffinic base oils combined with a sulphur additive is studied in the second part. The nature of the tribofilm created at the bottom of the threads formed during the second part of the study is characterized by X-ray Photoelectron Spectroscopy (XPS). The aim is to associate the surface analysis results with the efficiency obtained during the form tapping tests.
The results show that the lubricant viscosity does not have any influence on the tapping torque values whereas physico-chemical interactions between different base oils and a sulphur additive can modify importantly the overall efficiency of lubricant.

1 Introduction

The tests of cutting fluids are used to improve the formulation of lubricants and consist in evaluating the behaviour of additives or new compounds. The technological development in machining has to be followed up by an evolution in the formulation of lubricants. The environmental aspect is not without importance. The ecological regulations for the lubricants become increasingly constraining. The reduction of certain toxic chemical substances is one of the reasons why cutting oil manufacturers are trying to develop new, more efficient but less toxic oils.
The efficiency evaluation is realized typically using laboratory tests such as the four-ball test, or with Timken and Reichert machines. These tests are easy to carry out and do not require a lot of time and material. The contact between the surfaces and the parameters used for the tribological tests do not reproduce exactly the tool-chip-workpiece zone, though. These tests are still however the fastest way to study the antiwear or extreme-pressure properties of lubricants. During the seventies, a new approach for testing lubricants under machining conditions was developed. The first attempts of cutting fluids evaluation by machining tests showed that these results can be quite different from those obtained by the tribological tests. Several authors believe that the tests in real conditions of machining are the only one that make it possible to evaluate the lubricants in a relevant way [1-5]. The disadvantages of the machining tests are principally: important quantity of material used, long duration and high cost of tests.

These tests require the rigorous control of several machining parameters so that the true effect of the lubricant can be studied (problem of repeatability of the tests). This difficulty is often overcome by the preliminary series of tests with a fluid of reference to take into account, and estimate, the effects of others parameters.
The aim of machining test evaluation is not to determine the properties of the lubricants but to establish which type of lubricants is the most appropriate for a given application and improve the quality of machining. The measurements of cutting forces and specific cutting energy become the main method of lubricant evaluation. These tests allow one to assess the lubricants in a fast and more reliable way compared to the tool life tests. This method is very advantageous because less expensive, requiring less material and characterized by a very good repeatability. It is thus understandable that this type of test is frequently used to evaluate lubricants in tapping [6,7], drilling, reaming or boring. The importance of the fluid during the operation of tapping was proven through several studies [8]. In order to evaluate the efficiency of the lubricant, we measure the tapping torque variation according to standard ASTM D5619. The performance of the lubricant increases with the decrease of tapping torque values. The use of small quantities of oils makes it possible to test several lubricants.
Form tapping is a method of manufacturing an internal thread which is different from cut tapping. The thread is obtained by displacement of the work material. There is no chip formation. Figure 1 presents the difference between the micrographs of cut and formed thread. The consecutive movement of lobes of a tap produces the thread. Each lobe provokes a three-dimensional plastic flow that leads to a strain hardening of the work material [9]. The consolidation of the matter (increase close to 100% of initial hardness) has a crucial impact on the tool wear.

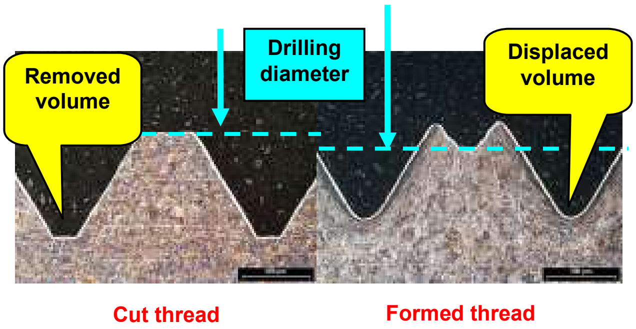

Figure 1: Micrographs of cut and formed thread

The formed threads are distinguished from those obtained by cut tapping, in addition to the absence of chip, by an increased tool life, an increased reliability and better geometrical and mechanical characteristics of threads [8]. That's why this process acquires greater importance for several mechanical industries such as the automotive sector.

This study is divided in two separate parts. Firstly, we are using the tapping test to consider the effect of three lubricants, having different viscosities but the same additives, on the variation of tapping torque. Secondly, the base oil effect is examined using the tapping torque test as well. Two different paraffinic base oils (chemically active and chemically non-active) with and without a sulphur additive are evaluated. The cut parts of formed threads are then characterized using X-ray Photoelectron Spectroscopy, in order to correlate the efficiency of the lubricant with the tribochemical reactions that took place on the surface of the threads during the form tapping operation.

2 Experimental materials

2.1 The tested lubricants

2.1.1 Lubricant viscosity effect

The three lubricants used in this part of the study are commercially available metalworking fluids formulated for severe machining operations of ferrous and non ferrous materials. They consist of a paraffinic base oil mixed with the additive package. One lubricant differs from any other only by its viscosity. Table 1 shows the available information about the tested lubricants, their viscosities and their designations used for this study.

Fluid designation used for this work	Lubricant viscosity and composition of fluids
F1	9 mm^2/s at 40°C – Paraffinic base oil + additive package
F2	23 mm^2/s at 40°C – Paraffinic base oil + additive package
F3	46 mm^2/s at 40°C – Paraffinic base oil + additive package

Table 1: Tested fluids and their designations – lubricant viscosity effect

2.1.2 Base oil effect

Four different fluids are tested in this part of the experiment. We examine the effect of two different mineral base oils: chemically active (containing sulphur components) and chemically non-active. In addition, two other blends are prepared – the tested base oils are combined with a sulphur additive (with the same concentration in both cases) (see Table 2). Sulphur additives are known to play a major role in different severe machining operations; in this particular case, the use of sulphur will moreover allow us to highlight any interactions with sulphur components from the chemically active base oil. Hence the choice of a sulphur additive.

Fluid designation used for this work	Composition and nature of fluids
F4	Chemically active paraffinic base oil
F5	Chemically active paraffinic base oil + S additive
F6	Chemically non-active paraffinic base oil
F7	Chemically non-active paraffinic base oil + S additive

Table 2: Tested fluids and their designations – base oil effect

2.2 The tapping test materials and experimental procedure

The tapping tests are carried out on a DMC65V (Deckel Maho Gildmeister) 3 axes vertical machining centre equipped with a 840D Siemens CNC. The forces generated during the form tapping operation are measured with a 9173 Kistler force transducer. This device records the force Fz and the torque Mz. The direction Z corresponds to the axis of the tapped specimen and the spindle of the machining centre. The use of tapping torque to compare the performance of different fluids is the normalised method ASTM D5619 standard test [7]. The system of acquisition includes a charge amplifier and a PC provided with a data acquisition card and Dasylab acquisition software. The fabrication of workpieces turned on a CNC lathe in C70 carbon steel is prior to the form tapping tests. We are using the M12x1.5 6HX taps for reasons of sensor capacity and to provide a typical example of industrial practice. They are made of high speed steel (HSS) enriched with cobalt and TiN coated. The taps and material used during the tests belong to the same production batches. The tools are cleaned with heptane in an ultrasonic bath.

Before starting the tests, all the pieces of machining equipment, including the force transducer with its protection case and the concentric installation, were carefully cleaned. After that, they were fixed on the table of the machine centre. The 20 mm-diameter cylinders previously machined on the CNC lathe were mounted in the concentric installation. Figure 2 shows the machining equipment used for tapping tests. The accurate hole diameter and concentricity have a considerable effect on the tapping torque value. A larger diameter means that there will be less matter to push back, therefore less energy for the tap to form thread, and consequently lower tapping torque and less remarkable difference between the tested lubricants. That is why the drilled and bored holes adapted to the dimensions of threads M12*1.5 are produced directly on the machining centre previous to the tapping test. The operations of drilling and boring are made with sulphurized water-based emulsion (concentration 5%). The diameters of bored holes are rigorously checked before each tapping operation. The holes are cleaned and then filled with test fluid. The tapping torque developed during each test is monitored and recorded.

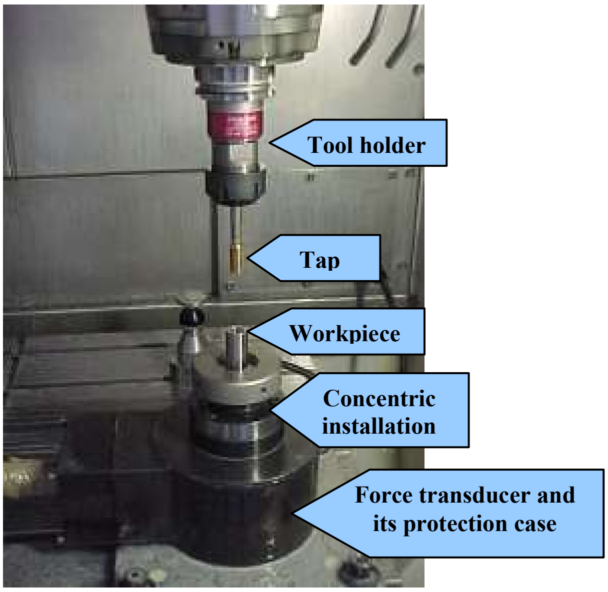

Figure 2: The set up for the machining operation

2.3 Surface analysis of tapped specimens

This part of the experiment aims at associating the presence of lubricant elements on the surface to the performance obtained during the tapping test. The surfaces of threads formed with three different fluids (F4, F5 and F7) are observed with X-ray Photoelectron Spectroscopy (XPS). XPS analysis is carried out with an Al K_{α} line excitation source. We are using a PHI Quantera SXM spectrometer. The residues of lubricants are ultrasonically removed from cut parts of formed threads in pure heptane just before their introduction into the analysis vacuum chamber. The analysis is made in to the pitch of the formed thread (thread root) because that is the zone where the most severe contact with the working tap occurs. Figure 3 presents the zone of analysis of the cut part of the formed thread.

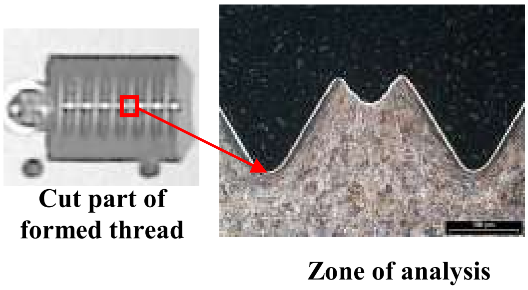

Figure 3: Analysed zone of the cut part of the formed thread

Afterwards, the thread surfaces are slightly etched with Ar^+ in order to remove the adventitious carbon (etching thickness 1,6 nm). The ion etching conditions are: 1 kV, 2 x 2 mm², 1.6 nm/min (we refer to SiO_2 etching).

3 Results

3.1 Viscosity effect in form tapping

The objective of this test is to compare products of different viscosity but having the same additives. Figure 4a and 4b presents the results of the tapping test with the three lubricants presented in chapter 2.2.1. A new tap is used for every lubricant. The graph 4a shows the tapping torque evolution as a function of time. Form tapping is a process where the cutting section is constant. It means that the tapping torque varies little when all the active lobes of tap are entered into the material to form the thread. The average tapping torque is evaluated when the thread formation takes place. This period of time corresponds to the quasi-stationary state comprised for this test between 1.5 and 3 seconds. The average tapping torque for the three lubricants reaches the value of about 30 N.m. It can be assumed that the F2 lubricant acts differently at the moment of tap entry into the material (0.5 s of time) after which it achieves the same tapping torque value as the two others fluids. The graph 4b sums up the results and gives the average tapping torque values obtained for every fluid from three repetitions. There is little difference between the results obtained for the three lubricants. This experiment confirms that viscosity has no influence on the efficiency of lubrication in form tapping and that it is in fact the additives which will determine the fluid efficiency. The hypothesis that the lubricant viscosity does not have any influence in form tapping was already mentioned elsewhere [8]. As a result, the rest of the experiment will be concentrated on the physico-chemical action of different base oils, combined or not with a sulphur additive, on the performance of a lubricant. An important part of surface analysis will follow the second part of the tapping tests.

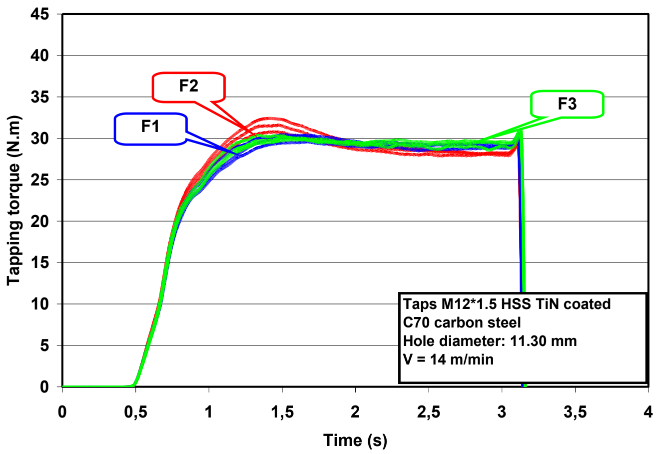

Figure 4a: Tapping torque evolution as a function of time for F1, F2, F3 lubricants having different viscosities

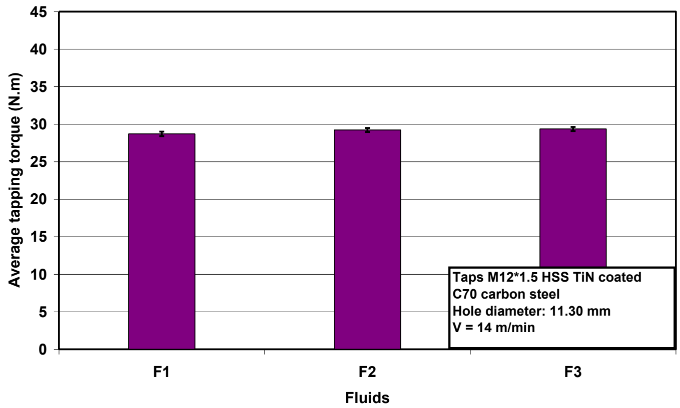

Figure 4b: Average tapping torque for F1, F2, F3 lubricants having different viscosities

3.2 Base oil effect in form tapping

The first section of the base oils tapping test aims at checking the variations between the taps used for this part of experiment. The dispersions can be due to the roughness and the microgeometry of the tools. We evaluate these differences in order to establish the correction coefficient of the taps. As a result of this coefficient, the tap effect on the latter results can be ignored.

Four new forming taps are employed (FT1 – FT4); each one is used to form four threads with the reference fluid (F4). All the taps have the same dimensions and belong to the same production batch. The first graph (see Figure 5a) shows the variation of tapping torque value for different taps. The second one (see Figure 5b) gives the average tapping torque value for every tap calculated from four tapped holes with the F4 reference fluid. The existing differences between the taps were evaluated to be of the order of 2%. The correction coefficient is calculated and applied for the second section of the experiment. The taps are carefully cleaned in heptane using an ultrasonic bath before the next use.

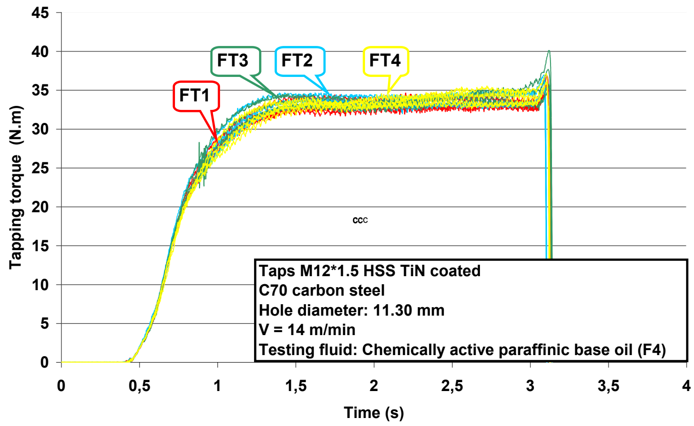

Figure 5a: Tapping torque evolution as a function of time for four new taps (FT1-FT4) with the reference fluid (F4)

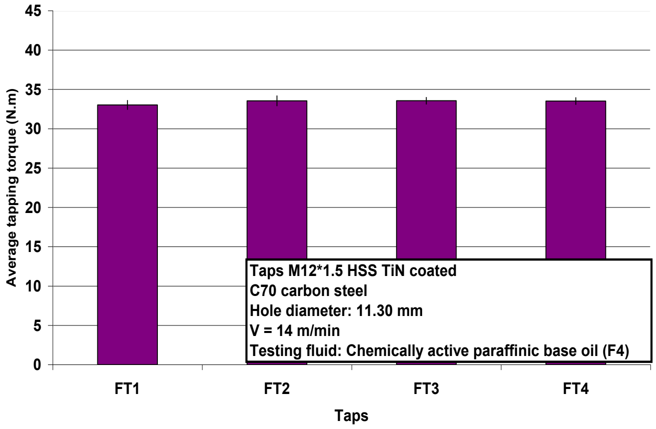

Figure 5b: Average tapping torque corrected for four new taps (FT1-FT4) with the reference fluid (F4)

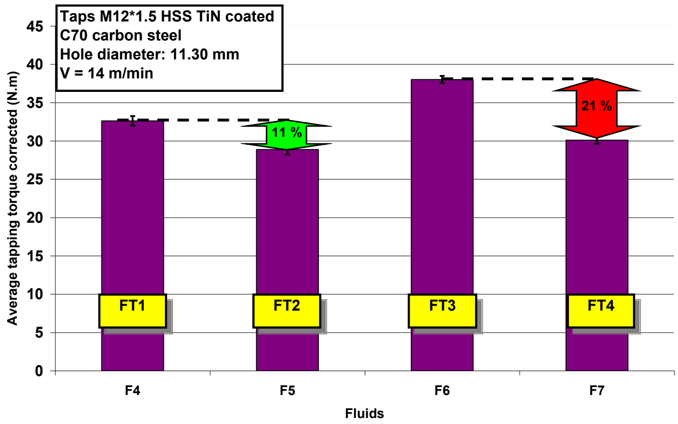

Figure 6b: Average tapping torque corrected for four (F4-F6) tested fluids (base oil effect)

The second section of this experiment consists in evaluating the performance of the four tested fluids (see 2.1.2. The tested lubricants – Base oil effect): F4, F5, F6 and F7. One tap is used to form five threads with the one and only tested lubricant. The evolution of tapping torque as a function of time (see Figure 6a) shows the differences between the tapping torque values for each fluid. The second graph summarises the results and presents the average tapping values, adjusted with the correction coefficient of the taps, for these lubricants (see Figure 6b). Among the tested fluids, F5 and F7 are the most efficient due to their lower average tapping torque value. The action of sulphur additive enables one to decrease the tapping torque by up to 21% in the case of F7 fluid compared to F6 and up to 11% in the case of F5 fluid compared to F4. The tapping torque value obtained with F7 fluid is comparable with the one obtained with F5, as a result we can deduce that base oil does not have any important effect on the final efficiency of the lubricant. It should be emphasised that the efficiency of the F4 fluid is higher than the one of F6, though. It is suggested from this study that the chemically active elements contained in the F4 fluid somehow compete with the sulphur additive for the surface and consequently prevent the additive present in F5 fluid from being as efficient as in the case of the F7 fluid. The formed threads with F4, F5, F7 fluids are cut for the purpose of surface analysis. XPS Spectroscopy is employed to show the chemical states of lubricant elements present on the surface.

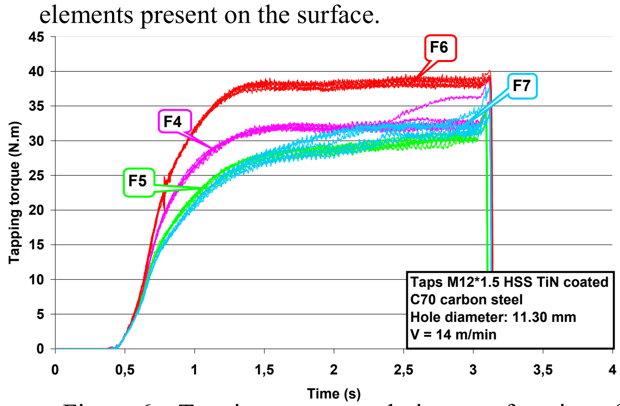

Figure 6a: Tapping torque evolution as a function of time for four (F4-F6) tested fluids (base oil effect)

3.3 Results of surface analysis of tapped specimens

Electron binding energies in the XPS spectra of threads formed with the three test fluids were calibrated against the carbon C1s main peak assumed to be 284,8 eV. The binding energy of C1s at 286,2 eV and 287,6 eV can be assigned to C-O, C-N and O-C-O, C=O bonds respectively. The nitrogen traces, coming probably from the tap coating, are detected on the surfaces. The C1s component at 288,8 eV can correspond to O-C=O bond. The binding energy of O1s at 529,8 eV can be attributed to the existence of different metallic oxides. The iron Fe 2p3/2 main peak at 710,5-711eV confirms the presence of oxides and probably of iron sulphides as well. There is possible interference of energy position between the peaks of these two products. Another O1s components at 531,4 eV and 532,8 eV can belong to O-C organic bonds and O=C, sulphates respectively. The sulphur peak S2p indicates the presence of two different components: sulphides and/or adsorbed sulphur species at 162,7-163,3 eV and sulphates at 168-168,6 eV. The decomposition of the sulphur peak detected on the thread surfaces obtained with the three tested fluids differs, though (see Figure 7). It can be noticed that on the surface of the threads obtained with:

- F4 fluid, the sulphur peak is composed by a majority of sulphates (component towards 168,4 eV) before ion etching and of sulphides or/and adsorbed sulphur compounds at 163,24 eV after ion etching
- F5 fluid, the sulphur peak contains two components: more intense at 168,35 eV – sulphates and less intense at 163,17 eV – sulphides or/and adsorbed sulphur compounds before ion etching and the same components of similar intensity at 167,93 eV and 162,67 eV after ion etching respectively
- F7 fluid, the sulphur peak contains two components of similar intensity at 167,93 eV – sulphates and at 163,17 eV – sulphides or/and adsorbed sulphur compounds before ion etching and the same components of similar intensity at 168,05 eV and 162,67 eV after ion etching respectively

Table 3 indicates the different proportion of sulphur components for these three lubricants. The chemical shifts of sulphur and proportions between two sulphur peak components after ion etching for different fluids will be the object of the latter discussion.
Table 4 shows the atomic percentage of major chemical elements detected on the surfaces of formed threads with the three tested fluids before and after ion etching. It can be reported that the surface of the thread formed with F4 fluid was etched sufficiently to remove the carbon layer and to reach the oxide layer (increased iron and oxygen atomic concentration). It suggests also that the tribofilms created with two other fluids (F5 and F7) can contain carbon.

	Sulphides or/and adsorbed sulphur compounds	**Sulphates**
F4 before ion etching	13	87
F4 after ion etching	100	-
F5 before ion etching	41	59
F5 after ion etching	54	46
F7 before ion etching	43	57
F7 after ion etching	47	53

Table 3: Different proportions of sulphur components on the sulphur spectra of formed threads with F4, F5 and F7 tested fluids before and after ion etching

Thread formed with the fluid	**C**	**O**	**Fe**	**S**
F4 before ion etching	65.5	29.8	3.3	**1.4**
F4 after ion etching	16.0	52.4	30.1	**1.5**
F5 before ion etching	65.1	30.9	3.2	**0.8**
F5 after ion etching	55.7	34.3	7.9	**2.1**
F7 before ion etching	65.5	30.2	2.9	**1.4**
F7 after ion etching	62.5	32.0	4.8	**0.7**

Table 4: Atomic percentage of chemical elements detected on the surfaces of formed threads with F4, F5 and F7 tested fluids before and after ion etching

4 Discussion

The surfaces of formed threads with F5 and F7 fluids are mainly composed of sulphates, sulphides or/and adsorbed sulphur compounds and iron oxides. We can deduce that the efficiency of lubrication brought about by these two lubricants is due to the sulphides probably to the iron sulphides. It is impossible however to specify from our data the exact chemical form of these species. The protective film of sulphides as a reaction product of sulphur compounds with a metallic surface is well known for very good anti-seizure and anti-welding behaviour in extreme conditions such as machining (high temperature, pressure). The sulphides can also be easily sheared. They may lower the friction coefficient between the surfaces, which could explain the advantageous effect on the forces during the form tapping operation [10,11]. The tribofilm on the surfaces of threads formed with F4 fluid seems to be composed of sulphides or/and adsorbed sulphur compounds on which the sulphates layer is deposited. The position of sulphur peak at 163,24 eV detected on the surfaces of formed threads with F4 fluid after ion etching could suggest the presence of another sulphide compounds instead of those detected on the surfaces of threads formed with F5 and F7 fluids (sulphur peak at 162,67 eV after ion etching) and/or the presence of adsorbed sulphur species. This is the possible explanation of the different efficiency of these three tested fluids. The friction behaviour of iron sulphides, for example of FeS and FeS_2 compounds, can be very different [12]. This observation is made referring to the chemical shift of sulphur peak before and after ion etching but it should be taken with caution because the ion etching can modify the oxidation number of sulphur atoms (reduction).
It is difficult to determine the role and the process of sulphate formation for the three tested fluids (sulphur additive decomposition product or/and sulphides oxidation product). It is inferred that sulphur elements coming from chemically active paraffinic base oil can compete with the sulphur additive on the surface. The same sulphur additive is twice more efficient in the chemically non-active paraffinic base oil. The efficiency of the two fluids containing the sulphur additive (F5 and F7 fluid) is comparable.

5 Conclusions

(1) The major role of fluid in form tapping is to lubricate by the physicochemical way. The viscosity does not have any effect on lubricant efficiency.
(2) The two most efficient lubricants form sulphides or/and adsorbed sulphur compounds, sulphates and iron oxides on the thread surfaces. The presence of sulphides is probably the reason for their good efficiency.
(3) The chemically active and chemically non-active paraffinic base oils combined with a sulphur additive provide similar lubricant efficiency. There is a possible competition between the sulphur elements coming from base oil and the sulphur additive.
(4) This study highlights the fact that the cost of fully formulated oil can be reduced by the use of a less elaborated base oil. The efficiency of a fully formulated oil depends on the activity of its additives.

a) BEFORE ION ETCHING b) AFTER ION ETCHING

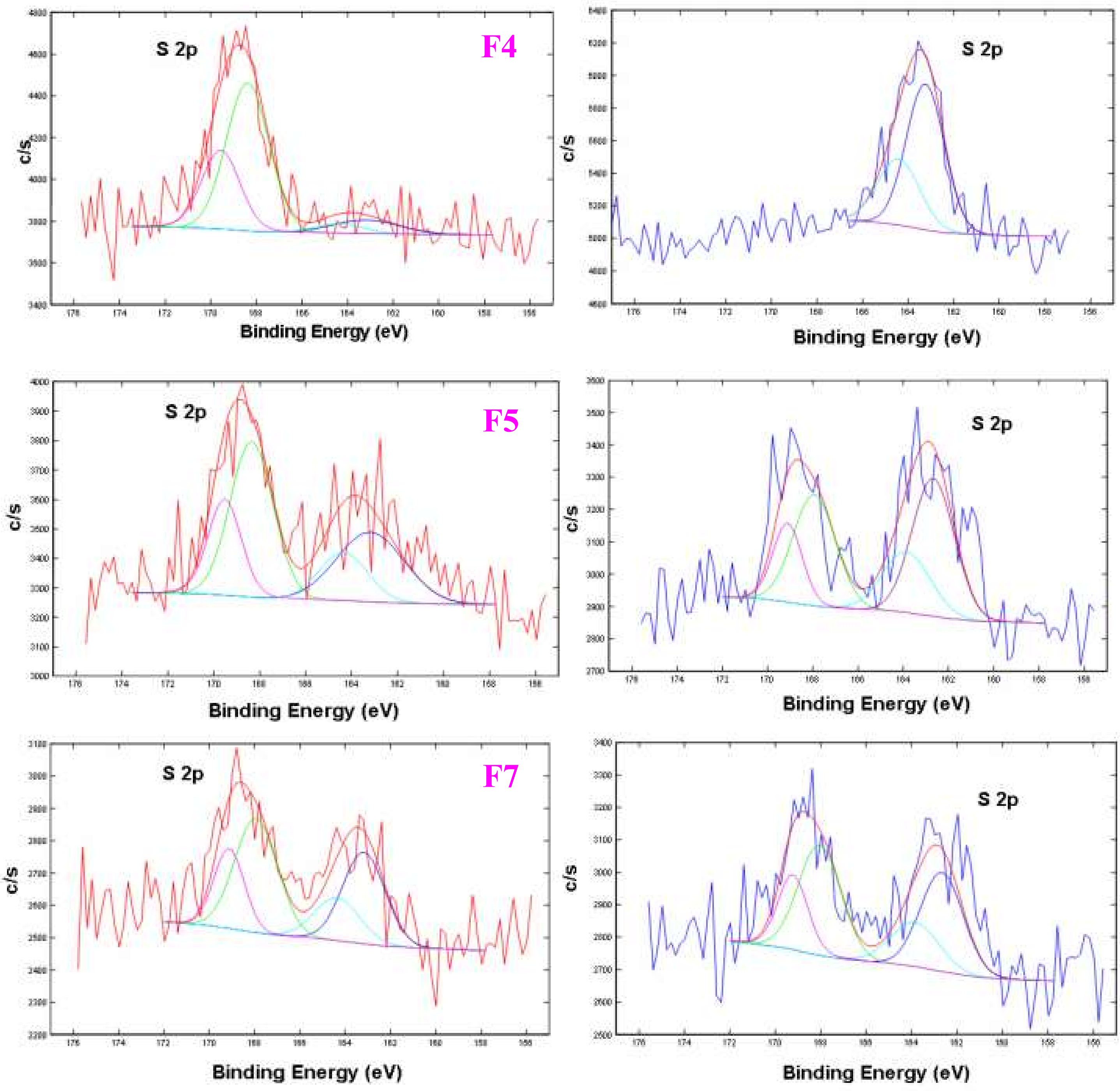

Figure 7: Chemical shift of sulphur peaks on the surfaces of formed threads with F4, F5 and F7 tested fluids

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