EFFECT OF FILM PLASTICIZING UNDER MULTISTAGE ULTIMATE STRAIN

Ata A. Miatiev* and Galina V. Khil'chenko

Pro Scientific & Technical Service, Prague, Czech Republic

ABSTRACT

The results refer to changes of mechanical properties of thin-walled nickel-chromium alloy tube specimens after multistage cold drawing. The goal was to analyze the possibilities of changing the kinetics of hardening and destruction under influence of deposited metal oxide films.

It has been noticed that under ultimate strain by drawing strength of coated tube specimens is much more than that of specimens without films. Specifically, the ultimate value of strain for coated tube specimens increases. At the same time the coated specimens have relatively high plasticity. The strength of coated specimens after ultimate strain is 14 – 23 % higher and plasticity is 4 times more. Specifically, that the ability of alloy to stand multistage ultimate strain can be increased more. It can be done by overlaying the film each time the specimen is drawn. In addition, there are the technological advantages of using the films under drawing. These advantages are the better quality of surface and decrease of drawing force. The possibility of effective application of plasticizing films to machining tools has been noted. The plasticity effect in this case is 50 – 70% of that when films are deposited on material. Besides, they work as solid lubricants, decreasing power consumption for working and wear of tool. In explosive stamping tests of titanium-base alloy coating with oxide films resulted in double increase of plasticity.

KEYWORDS

Film plasticizing, oxide films, ultimate strain, drawing, stamping, nickel–chromium alloy, films in metal working, solid lubricants.

INTRODUCTION

Amorphous oxide films, which are ideal elastoplastic bodies, are capable of essentially improving the plastic strain uniformity in the surface layers of engineering alloys under out-of-contact strain during mechanical tests [1]. Study of the effects caused by such films on the properties of alloys in working (drawing, rolling, stamping, extruding, and others) is of enormous interest for both science and practice. The process itself is of equal importance.
RESULTS

There is a special program for relevant research. This program involved: – drawing of thin-walled tubes, both uncoated and coated, through dies of various diameters without in-process and finish anneals; and– mechanical tests of the tubes.

The drawing conditions were standard, with spindle oil used as lubricant. All specimens of a nickel–chromium alloy were cut from one batch of as-received tubes 7.0 x 0.3 mm in diameter. The following sets of specimens were tested.

**Set A.** As-received (initial) tubes. The test procedure was the following: As-received tubes were drawn sequentially through dies of different diameters. After each drawing, three specimens were chosen from the set. The cross-sectional strain (the ratio of the reduction in the initial tube diameter to the inner diameter of the die) was controlled. These specimens were subjected to tensile tests at 573 K.

**Set B.** As-received tubes were coated by amorphous zirconia to a coating thickness of 1.5 µm. The drawing procedure, specimen selection, and mechanical tests were identical to those applied to Sets A.

**Set C.** Set B tubes, each time after being drawn but before being tested, were additionally coated by amorphous zirconia to form a 1.5-µm-thick film. The subsequent testing procedure was as in other sets.

We were also interested in achieving the highest possible reduction of the tube area.

The mechanical test data were processed in terms of ultimate tensile strength, offset yield strength, and total strain at fracture versus prior drawing strain (reduction of area).

Figures 1-3 present the results from mechanical tests at 573 K of tubes drawn to various extents. Tested were pieces of Set A (uncoated) tubes, Set B (zirconia-coated) tubes, and Set C tubes (Set B tubes with a zirconia coating overlaid after each drawing).

![Figure 1](image1.png)

**Fig.1:** The plot of the 573 K ultimate tensile strength vs. reduction of area after drawing for nickel–chromium alloy tubular specimens of Sets A, B, and C. The film is zirconia 1.5 µm thick.
Fig. 2: The plot of the 573 K offset yield stress vs. reduction of area after drawing for nickel–chromium alloy tubular specimens of Sets A, B, and C. The film is zirconia 1.5 µm thick.

For uncoated materials, the offset yield stress and ultimate strength increase to attain the maximum value when the reduction of area is 42%, then decreases; when the reduction of area is larger than 54%, the specimen is broken in drawing, being lengthwise separated into fibers. The yield stress values...
for zirconia coated specimens are lower than for uncoated specimens. In addition, there is no peaks in the curve of Fig. 2, and the coated specimens do not break when the reduction of area is 60%. Of special interest are the results from zirconia-coated specimens on which zirconia was overlaid after drawing. For the reduction of area from 6 to 28%, the yield stresses in the coated specimens differ little from similar values in zirconia-coated Set B specimens. For the reduction of area larger than 28%, zirconia overlaying produces a substantial increment in the yield stress as compared both to the uncoated specimens and to the Set B specimens. The same trend is observed in the ultimate tensile strengths for the Set C specimens.

In this way, the application of zirconia to Ni–Cr tubes promotes hardening in work, and overlaying after each drawing operation considerably strengthens this effect. Characteristically, all deposited films gradually increase the alloy plasticity (Fig. 3). While the total elongation at ultimate strain hardening in drawing of the uncoated alloy is within 1%, its value for a coated alloy is substantially higher. Specifically, the elongation amounts to 4% for the specimens coated by zirconia before being repeatedly drawn. And for specimens on which zirconia films were overlaid each time they were drawn, the elongation is 6%.

The following questions arise: What is the reason for this? And which is the expression of this in terms of materials science?

Prior research, specifically, metallographic evidence, implies that the effects of amorphous deposited films are a consequence of the same physical processes in materials that were discovered earlier in mechanical tests [1]. The only distinction is that the role of the surface in working and under the contact action of the extrusion device becomes more significant. For example, under the contact action during drawing, films (that naturally possess a certain set of properties) can efficiently improve the uniformity of micro plastic strain in polycrystalline materials. Their effect in contact action becomes much stronger. As a result of the increased uniformity of plastic strain in every grain of the polycrystalline material (it was noted that additional crystallographic directions were involved in this [1]), the deformation defect structure becomes far more homogeneous and energy-balanced. A situation appears where grain boundaries; segregations (inclusion phases); the second, higher strength phase; texture; and other structural details of the initial material substantially loss their tendency to induce various local processes during deformation. Most likely, the weaker tendency of the polycrystalline material to locally accumulate structure defects is precisely the reason for the increment in the integral strength simultaneously with a relative increase in plasticity at ultimate strain hardening.

The results of the tests and studies on this set of specimens should be regarded as extraordinary, even if the effect of films on the mechanical properties [1] is taken into account.

Firstly, the films were applied on specimens before drawing. The observation that the effect characteristic of each type of coating is retained in multiple-draft (!) drawing is unexpected and very important in practice.

Secondly, the films after multiple-draft drawing substantially add to the plasticity at the ultimate extent of hardening (reduction of area). Note that the tests did not involve anneals, which is important.

Thirdly, not only the deposited films were strained together with the tube, but they were (at least, the film on the outer tube surface was) in direct contact with the extrusion device (die). However, the films each retained their distinctive effects on the mechanical properties of tubes in multiple-draft drawing. This provides grounds to suggest that not only are films conserved on the tube surface, but they even cause a certain influence on the mechanical properties of the substrate metal. Unlike the ordinary conditions of mechanical tests, there is a direct action of the extrusion device in this case. Therefore, the film may be regarded as a solid lubricant.

Lastly, the fact itself that the strength and plasticity increase in tandem in drawing is evidence that films are capable of substantially changing the conditions of contact micro plastic strain of the substrate metal. The most significant consequence of this is the lowering of the critical level of the deformation defect density in the substrate (the level that causes fracture of the material). The material after being drawn has a more perfect structure regarding its mechanical properties.

Based on experimental evidence (especially, from the sets of specimens on which an oxide film was overlaid each time they were drawn), we infer that the ability of the oxide coating to inhibit premature surface breakdown of the substrate is the dominant. In other words, the film creates a situation such that the surface layer remains more plastic than the metal bulk even under ultimate strain hardening. In this regard, the film
together with the surface layer of the substrate acts as an efficient solid lubricant in the device–metal system. The fact that such a lubricant can work under the increasing strength of the workpiece seems most significant. The possibility of surface softening due to the deposited film in metal working with a superhard contact action of the device is of enormous value for the engineering. This concerns not only rolling, stamping, drawing, and extruding, but also cutting, milling, drilling, etc. In addition, the surface softening of one of the partners of the contact pair can aid in solving many problems in tribology (friction and wear). Oxide films might find extensive and diversified application in metal working. When the yield strength is reduced, especially in difficult-to-extrude alloys, energy requirements in metal working may be considerably reduced. The feasibility of reducing temperature and strain load goes along with this. The increment in plasticity makes it possible to attain deeper deformation in high-strength and low-ductility alloys, as well as an increment in the ultimate strain, which is usually controlled by work hardening and requires in-process anneals. The main conclusion from our tests and studies, which is very important in practice, is that the optimal effect of coatings in tube drawing, as assessed by the reduction in the drawing force, can reach 40–70% depending on the alloy and drawing conditions. Other achievements are high surface quality, structural homogeneity, and a set of mechanical properties. In particular, the scatter in the mechanical properties of coated tubes after drawing under the optimal conditions is reduced several times. When drawing small-diameter thin-walled tubes, comparative tests of the efficiency of deposited oxide films were made on instrumental steel filaments. Pieces of stainless steel and a chrome–nickel alloy were drawn. The following control parameters were used: drawing force, chemical composition of the die surface, and state of the die surface as observed under a scanning electron microscope. Drawing with and without lubricants was performed, and various deposited oxide films were tested. This study showed the following results.

The general trend of the effect caused by films on the drawing force during unlubricated drawing fits the following pattern (regarding the reduction in the effect, all other conditions being equal):

- amorphous oxide films >> crystalline oxide films,
- multilayered films > single-layer films,
- films on the tube and die > films on the die.

In addition, the drawing force versus film thickness relationship has the trend plotted in Fig. 4 and is characteristic of all film materials tested. The largest reduction in the drawing force on the die for various films ranges between 5 and 30%. It was concluded that, if the film is applied only to the tool, a softening effect, like that produced by the film on the workpiece, is observed. However, in this case, surface softening of a work or cut metal is 30–50% lower.

![Fig. 4: The effect of thickness of the deposited film on the drawing force](image)

The general trend in the effect caused by films on the drawing force during lubricated drawing with organic lubricants (regarding the weakening of the effect, all other conditions being equal) depends strongly on the lubricant and film structure. Characteristically, all other conditions being equal, the drawing force varies as a function of film thickness as plotted in Fig. 4. The strongest effect is achieved in the case where films are applied to both the tube and the die. In contrast to dry drawing, the crystal and layered structure of the film are weaker factors in this case. We concluded that with liquid lubricants the ability of the film to retain the lubricant is of primary importance. In tests, the initial reduction of the
drawing force was as high as 50–70%. However, this effect weakens to the vanishing point with increasing the operation time of the die. An analysis of the chemical composition of the die and periodic examination of the surface with an electron microscope showed that, for any drawing conditions and material, there is mass transfer of chemical elements from the workpiece to the die surface. Under heavy drawing conditions with an uncoated die, mass transfer occurs in the first centimeters of drawing even if a lubricant is used. When there is a film on the die, the elapsed time (the number of meters drawn) until mass transfer to the working surface becomes evident increases by several orders of magnitude. Selective contamination of the film by the chemical elements from the workpiece is still observed, but the film continues operating until completely fretted.

Tests and study showed that for almost any working tool material and any work alloy, a film with optimal efficiency and wear resistance could be created. The practicality the wearlessness principle due to periodic renewal of the film is enormous. It has been noted that the higher the strength, hardness, and wear resistance of the tool material, the higher the efficiency of the periodically renewed coating.

Fig.5: An item manufactured by explosive stamping from a low-ductility titanium-base alloy coated with a plasticizing film

Another revealing example of the efficiency of softening films was a comparative explosive stamping test on titanium-base alloys. Sheet pieces were tested for manufacturing ring stampings with an overall strain of 12% at a strain rate of 7500 m s⁻¹. Previously, a high-ductile titanium-base alloy (1.5Al-1.0Mn; σ = 600 MPa, δ = 20%) was used to manufacture these stampings. There were no problems in one-stage stamping of this alloy. The titanium-base alloy (6.0Al-2.0Zr-1.0Mn-1.0V), which has higher strength but lower ductility (σ = 1000 MPa, δ = 6%), is evidently preferable in terms of performance. However, with this substitution, cracking and warping were observed during stamping, even under incremental loading. The application of a softening oxide film to the original titanium sheet resulted in a defect-free stamping, which is shown in Fig. 5.

References