John F. Paciorek, P.E.
JFP Technical Services, Inc.

Low Friction Chromium......
Good Medicine for Ailing Surgical Instruments

Introduction

Low friction chromium (LFC) is an extremely hard and wear-resistant coating that can be deposited on precision metal instruments for improved performance. In the Spring issue of BONEZone, Part I of this article discussed the physical properties of low friction chromium (LFC) that make it an attractive and logical choice for coating of stainless steel instruments.

Part II addresses the physical makeup of LFC that affords it the inertness, which enabled it to pass the required laboratory tests in order to achieve USP Class VI approval and establish ISO 9000 biocompatibility; Part III, also printed in this article, discusses some of the field tests that were performed with LFC-coated stainless steel.

Part II: USP Class VI Test Results

Although high hardness and excellent wear resistance are two of the hallmarks of low friction chromium (LFC), the feature which completes the bridge to its acceptance as a coating for use in medical instruments is its inherent passivity in many types of chemical environments. It has long been known that a self-healing oxide layer of chromium can be formed simply by its exposure to air or by immersion in room temperature oxidizing acids.

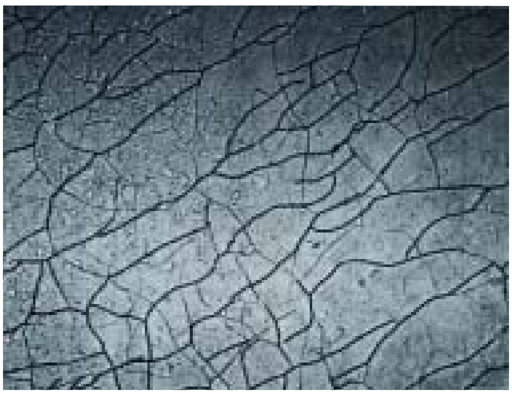

Figure 1. 400 X Magnification

Figure 1. Photomicrograph of the surface of a low friction chromium coating over a panel of type 304 austenitic stainless steel to illustrate the fine-grained nature of the microcracks on the surface inherent in LFC.

Figure 1 is a high magnification photomicrograph of the surface of a panel of AISI 304 stainless steel coated with LFC to illustrate the fineness of the microcracks that are characteristic of this coating. This compares to Figure 2, which is a photomicrograph of the surface of a conventional chromium coating. It is the fineness of these microcracks, which also affords LFC its improved corrosion resistance over conventional chromium by providing a more discontinuous path from the outer surface of the coating to the inner for invading fluids.

Figure 2. 400X Magnification

Figure 2. Photomicrograph of the surface of a conventional chromium coating over a panel of type 304 austenitic stainless steel to illustrate the coarse-grained nature of the microcracks on the surface relative to that of LFC.

This difference can be seen graphically in Figures 3 & 4, which are photomicrographs of cross-sections of both coatings that have been subjected to a Knoop microhardness impression under a 500-gram load. The 500-gram Knoop load can be seen to have caused gross propagation of microcracks in the conventional chromium coating, while the fineness of the micrograin of the LFC coating has allowed the microcracks to remain localized under the same loading.

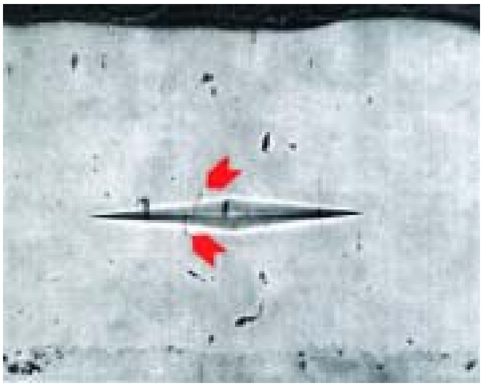

Figure 3. 800X Magnification, Unetched

Figure 3. Photomicrograph of a cross-section of a low friction chromium coating over type 304 austenitic stainless steel. The arrow points to fine microcracks in the immediate area of the impression that not have propagated beyond the impression into the balance of the coating.

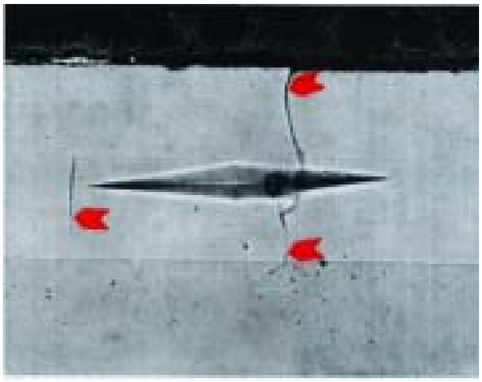

Figure 4. 800X Magnification, Unetched

Figure 4. Photomicrograph of a cross-section of a conventional chromium coating over type 304 austenitic stainless steel. The arrow points to microcracks that have propagated from the immediate area of the impression to the outer edges of the coating.

Internal Environment of the Human Body

The internal liquids of the human body can present a very hostile environment to medical instruments. Cellular fluids containing high concentrations of chlorides and organic acids can be very corrosive to uncoated austenitic stainless steel. While the normal pH of the body liquids is about neutral (pH 7.2-7.4), these values can rise to highly acidic (pH=4.0) at sites of bodily injury to highly basic where infections may be present. The use of invasive devices into this wide variety of aggressive chemical conditions can deem many other materials inappropriate, including chromium steels, low-grade austenitic stainless steels and sensitize stainless steels.

While it must be made clear that chromium coatings are not recommended for long-term use in the body, such as for implants, both field trials and laboratory-accredited testing have proven LFC-coated stainless steel medical instruments to be sufficiently impervious for shorter-term invasive uses, such as for surgical preparation of the receptor bone for the implant or for use in arthroscopic surgery, as results from the following tests illustrate:

USP Class VI Certification Testing

In order to evaluate the inertness of LFC coatings, panels of type 304 stainless steel were coated and subjected to the bank of tests dictated by the USP (United States Pharmacopoeia) for possible qualification for Class VI certification. Table I contains an itemized list of the tests performed, including all of the parameters of

continued on page 44

TABLE 1: USP CLASS VI BIOLOGICAL COMPATIBILITY TESTS PERFORMED AT NAMSA
TEST ARTICLE: AISI TYPE 304 STAINLESS STEEL COATED WITH LOW FRICTION CHROMIUM

Type Test	Extract Solutions Tested	Application Method	Test Length	Endpoint Criteria	Final Results
Acute Systemic Toxicity	a) .9% saline b) alcohol in saline c) polyethy glycol d) cottonseed oil	intravenous injection (5 mice, single dose)	3 days	mortality, weight loss, lethargy, convulsions, prostration, hyperactivity	a) No system toxicity b) No system toxicity c) No system toxicity d) No system toxicity
Acute Systemic Toxicity	a) .9% saline b) cottonseed oil	intravenous injection (5 mice, single dose)	3 days	edema, erythema	a) No negative reaction b) No negative reaction
Intracutaneous Toxicity	a) .9% saline b) alcohol in saline c) polyethy glycol d) cottonseed oil	injected under skin (2 rabbits, 5 places)	3 days	edema, erythema	a) No irritation or toxicity b) No irritation or toxicity c) No irritation or toxicity d) No irritation or toxicity
Intracutaneous Toxicity	a) .9% saline b) cottonseed oil	injected under skin (2 rabbits, 5 places)	3 days	edema, erythema toxicity, irritation	a) No negative reaction b) No negative reaction
Surgical Muscle Implantation	Medcoat article (10mm x 1 mm)	injected into muscle (2 rabbits, 4 places)	5 days	capsuling or irritation	No negative reaction
Surgical Muscle Implantation	Medcoat article (2mm x 9 mm dia)	injected into muscle (2 rabbits, 3 places)	9 days	capsuling or irritation	No negative reaction

NOTE: Detailed results of these studies are available from the Norman Noble Company, Cleveland, Ohio.

Low Friction Chromium, continued from page 43

the individual tests. All tests were performed under the auspices of NAmSA, North American Science Associates, Inc, an FDA-registered test agency.

For a clearer understanding of the essence of the results of these tests, they can be broken down into two basic categories, as displayed in Table 1: (1) four standard tests for toxicity wherein the test article (304 stainless panel coated with LFC) was extracted in various solutions prior to injection into the laboratory animal, and (2) two surgical muscle implantations wherein a sample of the actual LFC-coated panel was injected or incised into the body of the animal.

Results for the twelve individual tests for toxicity after three days showed that no evidence of any systemic toxicities was observed, nor any evidence of localized animal tissue reactions, irritation, edema or erythema, as a result of having been in contact with the extract solutions.

Results shown at the bottom of Table I for the two implantation tests after five and nine days showed no evidence of any negative reactions in the area of the implantation, such as capsuling or irritation, as a result of having an LFC-coated stainless steel article implanted under the skin.

Meeting of all of the above mentioned test criteria qualified LFC coatings for Class VI certification.

ISO/Tripartite Biological Compatability Tests

Various tests were chosen for testing LFC for its biocompatibility under a variety of test criteria. Table II is an itemized list of the tests performed, including all of the parameters of the individual tests. All tests were performed under the auspices of NAMSA, North American Science Associates, Inc.

Each of the tests listed measured the effects of an extract solution of the test article (304 stainless panel coated with LFC) under different test conditions, including application method, time length of test and endpoint criteria.

The last column of the table for the diversity of categories chosen shows that the solutions extracted from LFC-coated 304 stainless steel proved to be non-toxic, non-pyrogenic, non-hemolytic, non-sensitizing, and non mutagenic.

Conclusion

Laboratory-accredited tests showed that low friction chromium has passed the rigorous test criteria necessary to achieve Class VI certification that essentially certifies it to be a viable protective coating on stainless steel medical instruments destined to be used for invasive procedures.

PART III: FIELD TESTING OF LFC

While stainless steels (304, 316, 17-4 ph, 440-c, etc.) are the preferred-use commodity in hospital environments because of their excellent durability and low maintenance, there are many instances where even their performance can be enhanced when complemented with another material. One such category of implements would include those which must be repeatedly given an autoclave treatment or subjected to chemical sterilization processes prior to reuse in surgical procedures.

Although the surface of stainless steels is self-passivating in ambient air with no concomitant discoloration, the high autoclave temperatures or chemicals utilized in sterilization can turn the bright gun metal gray surface to a yellowish hue which, to many, is not very aesthetically pleasing in the hospital environment. More importantly, the discoloration can obscure laser markings of vital information such as instrument measurement graduations, company logos and part and model numbers.

TABLE 2: ISO/TRIPARTITE BIOLOGICAL COMPATIBILITY TESTS PERFORMED AT NAMSA
TEST ARTICLE: AISI TYPE 304 STAINLESS STEEL COATED WITH LOW FRICTION CHROMIUM

Type Test	Solutions Tested**	Application Method	Test Length	Endpoint Criteria	Final Results
Cytotoxicity	5% solution MEM elution-MG023	soak L929 mouse fibroblast cells	24 hrs.	CTE toxicity for confluent monolayer, vacuolization, crenation, swelling & % cellular lysis	NON-TOXIC
Rabbit Pyrogen	.9% saline	intravenous injection (3 rabbits, single dose)	3 hrs.	check for rise in rectal temperature every 30 minutes	NON-PYROGENIC
Hemolysis Test In Vitro	.9% saline	mix with .2 ml. rabbit blood	1 hr.	absorbancy as % hemolysis	NON-HEMOLYTIC
Delayed Contact Sensitization Test	.9% saline	intradermally injected= +occlusively patched=	7 days +13 days	delayed dermal reaction	NON-SENSITIZER
Ames Mutagenicity Study	.9% saline	added to slides with salmonella typhimurium	48-72 hr. incubation	screened for mutagenicity	NON-MUTAGENIC

*NOTE: Detailed results of these studies are available from the Norman Noble Company, Cleveland, Ohio.
**NOTE: All solutions were made by extracting the test article (LFC-coated 304 stainless) for various times.

Field Trials with Cyclic Autoclave Sterilization

Field trials were held to evaluate the relative propensity of uncoated and LFC-coated stainless steels to oxidize or discolor after having been subjected to repeated autoclave and chemical sterilization processes. Table 3 is a summary list of these tests, including the pertinent details of each trial and the endpoint criteria.

TABLE 3: PERFORMANCE TESTING OF LOW FRICTION CHROMIUM IN STERILIZATION

Type Test	Solutions Tested	Application Method	Test Length	Endpoint Criteria	Final Results
Autoclave exposure	low friction chromium on 304 SS	cyclic steam @ 121 degress C.	40 cycles (33 hrs. total)	visual & microscope for discoloration, rust	NO DISCOLORATION OR COATING DEGRADATION
Autoclave exposure sensitization test	low friction chromium on 304 SS	cyclic steam @ 132 degress C.	22 cycles	visual & microscope for discoloration, rust	NO DISCOLORATION OR COATING DEGRADATION
Autoclave exposure sensitization test	low friction chromium on 17-7 pH SS	cyclic steam @ 132 degress C.	22 cycles	visual & microscope for discoloration, rust	NO DISCOLORATION OR COATING DEGRADATION
Autoclave exposure sensitization test	uncoated 304 & 17-7 pH SS rods & panels	cyclic steam @ 132 degress C.	22 cycles	visual & microscope for discoloration, rust	YELLOW DISCOLORATION NOTED ON BOTH COUPONS
STERIS Cyclic sterilization	.0005" low friction chromium on 304 SS	"STERIS SYSTEM 1" chem process	100 cycles @ 140 degress C.	visual inspection for general appearance	COMPATIBLE WITH CHEMICAL PROCESS
H202 + plasma sterilization	low friction chromium on 304 SS	a) "STERAD" sterilization process	100 cycles @ 40 degress C.	visual appearance and compatibility	a) COMPATIBLE: NO COATING DISCOLORATION
H202 + plasma sterilization	low friction chromium on 304 SS	b) "STERAD" efficacy test	14 day incubation	bacterial growth	b) NO BACTERIA GROWTH

*NOTE: Detailed results of these studies are available from the Norman Noble Company, Cleveland, Ohio.
"STERIS SYSTEM 1" is a trademark of Steris Corporation.
"STERAD" is a trademark of Advanced Sterilization Products.

The first and second items listed in the table are autoclave tests. In the first test only LFC-coated type 304 stainless steel panels were tested and showed no evidence of discoloration after 40 cycles (33 total hours) at 121°C. For the second test a comparison was made of both uncoated type 304 stainless steel rods and panels and those coated with LFC; the same was done for type 440-C stainless steel rods and panels. Results showed that all of the uncoated 304 and 440-C stainless rods and panels appeared yellowish after 66 cycles, whereas all of the LFC-coated rods and panels still retained their satin-gray luster. Figure 5 is a side-by-side photograph of the uncoated and coated 440-C stainless rods after only twenty-two (22) cycles at 132°C to illustrate the difference.

Performance of LFC in Chemical Sterilization

Chemical sterilization tests were performed on LFC-coated stainless steel panels to complement the laboratory biological compatibility tests that had been performed essentially at room temperature for Class VI certification (See Part II). The intent of this testing was to establish whether or not the low friction chromium coating afforded 304 stainless steel any oxidation protection from high temperature sterilization processes which incorporate chemical additives.

Sterilization processes chosen for these trials included the Steris "Steris System 1™" which incorporates their own proprietary chemical additive and the Advanced Sterilization Products "Sterrad™ Sterilization System" which is also a unique process. These results are shown as the last three entries listed in Table 3. Results show that LFC-coated 304 was compatible with both the Steris System 1™ and the Sterrad™ processes, as no discoloration was noted on the test panels upon conclusion of the tests.

Conclusions

These test results which illustrate the high durability and excellent chemical resistance of LFC-coated medical instruments in autoclave and chemical sterilization environments show how even the fine, inherent qualities of stainless steel can be improved upon in some end use applications. Product enhancements due to LFC coatings include (a) resistance to yellow discoloration which might otherwise obscure vital information which is laser-marked on the instrument, including measurement graduations, logos, part numbers, etc., (b) increased wear- and scratch-resistance which can lead to longer tray and instrument life, and (c) maintenance of an original satin-gray finish which is apropos to a hospital environment.

Future Applications

A summary of the features of LFC are listed below at the end of this article. Some of these were effective in helping to achieve the results seen in this article, but others would suggest that additional uses may be found for LFC in the medical field. One obvious area which may hold promise is for coating cutting tools used in the preparation of bones prior to receipt of the implant. The features of LFC coatings relevant to this end use include (1) the low coefficient of friction of chromium which would help minimize packing of the bone mass in the flutes, leading to less heat buildup and thus decrease the chances for necrosis of the bone, (2) the high hardness and wear resistance of the coating which would afford longer tool life, and (3) the excellent adherence of the coating to the base metal for tough-drilling applications.

continued on page 47

SUCCESS STARTS WITH COMMITMENT!

Precision Metal Products, Inc. is an ISO 9001:2000 Certified company offering optimum quality forgings and machined products to the medical, industrial and aerospace markets. We are leaders in developing, maintaining, and completely integrating state of the art technologies at every level of the manufacturing process. A proven track record since 1963 is your assurance of exceptional product quality and unsurpassed customer service.

PMP uses a wide range of materials that include titanium, cobalt chrome molybdenum, aluminum, and stainless and high temperature steels. We also forge customer - specific proprietary alloys.

PMP

PRECISION METAL PRODUCTS, INC.

EXPERIENCE - DIVERSITY- GROWTH THROUGH INNOVATION SINCE 1963

Tom Cash, President • email: tomc@pmp-elcajon.com
850 W. BRADLEY AVENUE • EL CAJON, CA 92020
www.pmp-elcajon.com • Phone: 619-448-2711 • Fax: 619-448-2005

Low Friction Chromium, continued from page 45

Hallmark Features of Low Friction Chromium

Low friction chromium is a Class VI certified coating that possesses the following attributes for use in the medical industry:

1) The high hardness of LFC-coated austenitic stainless steels improve abrasion and wear resistance for general durability.
2) Excellent wear resistance helps tools maintain a sharp edge for surgical procedures.
3) LFC coatings can be applied to precision instruments without fear of distortion of the work piece.
4) LFC coatings can be applied as a final process, thereby negating the need for subsequent machining.
5) LFC coating thickness can be precisely controlled for different end uses.
6) LFC coatings are applied uniformly to all surfaces, including recesses and hole cavities.
7) LFC coatings resist oxidation and discoloration and maintain their luster even after cycles of high temperature autoclave treatment.
8) LFC coatings resist chloride-pitting corrosion common to uncoated stainless steels.
9) LFC coatings adhere tightly to the base metal.
10) LFC coatings have a much lower coefficient of friction than steel and most other metals, which can help decrease heat generation in applications involving sliding contact of two surfaces.

Figure 5, Actual Size

Figure 5. Photograph of the type 440C stainless steel rods that had been subject to autoclave sterilization for 22 cycles at 132°C (270°F). The upper rod is uncoated 440C and has developed a yellowish hue. The lower rod has been coated with low friction chromium and still retains its satin-gray luster.

Editor: John F. Paciorek is a registered professional engineer in the state of Ohio and is president of JFP Technical Services, Inc, an independent metallurgical testing laboratory in Mentor, Ohio.