

MATERIAL AND SURFACE FINISH ENHANCEMENTS

Introduction

In response to the ongoing trend in the semiconductor industry for increased surface cleanliness and improved surface chemistry in all gas supply instrumentation, improvements to existing stainless steel material and surface finish enhancements had to be developed. The material chosen to provide these improvements is 316L V.A.R. (Vacuum Arc Remelt) stainless steel. V.A.R. contains substantially less non-metallic impurities than are present in standard 316 or 316L.

Vacuum Arc Remelting is an additional refining process that removes non-metallic impurities from the base metal. These impurities, if in contact with semiconductor process gases, can be detrimental. They may react and form process contaminants.

The Mass Flow Controller's (MFC) internal surfaces have been further enhanced through the development of several new surface finish processes. These processes include modified machining techniques such as abrasive flow machining, micropolishing and electropolishing. The result of these process improvements is that surface foreign particle retention and/or contaminant release to the process is greatly reduced. The purpose of this paper is to summarize the enhancements Brooks Instrument engineers have developed by describing the material and surface processing changes and providing supportive evidence to verify the effectiveness of these changes.

316L V.A.R. Stainless Steel

The material from which most MFC's are fabricated is 316 stainless steel. This material is an austenitic stainless with a high content of chromium, nickel and molybdenum. These alloying elements give 316 stainless steel excellent corrosion-resistant properties. It has been the standard material used in semiconductor gas supply equipment for many years. As the industry requirements for better leak integrity have increased, the equipment suppliers have responded by offering metal sealed MFC's. In order to limit the number of metal seals required to seal an MFC, many of the joints, which previously used elastomeric seals, are now welded. Welding 316 stainless steel can degrade its excellent corrosion-resistant properties because of the relatively large carbon content (.08% max.). As the weld area cools, the carbon can deplete the alloy of seventeen

times its own weight of chromium while forming complex chromium carbides along the grain boundaries. These carbides do not exhibit the same corrosion resistance as the base metal and will lead to intergranular attack by some corrosive fluids. This problem has been eliminated by the use of 316L stainless steel which contains .03% carbon maximum. This reduction in carbon enhances the corrosion resistance by reducing the formation of chromium carbides.

Limiting the carbon content was a good first step. However, a higher purity 316L stainless steel with fewer non-metallic inclusions was desired. Non-metallic inclusions which are present in the alloy will degrade the material's corrosion resistance. If attacked, these inclusions could contaminate the process. When dissolved, these inclusions will leave a void which could entrap particles or retain moisture.

There are many types of non-metallic inclusions. The major types include: sulfides, aluminas, silicates, and globular oxides. These inclusions are almost totally removed from the 316L stainless steel through use of a process called vacuum arc remelt (V.A.R.). This process involves remelting the steel under vacuum. The dissolved gases, which create many of the inclusions, are removed under vacuum. One of the most important secondary alloying elements in 316L V.A.R. is Sulfur. To achieve good weldability and machinability, the alloy must contain some sulfur. In the 316L V.A.R. a sulfur content has been selected which is just high enough to maximize weldability and machinability while limiting the formation of sulfides.

With these material enhancements, a cleaner, more homogenous and better corrosion resistant alloy is obtained.

Internal Surface Enhancements

The narrowing of semiconductor feature line width down to the sub-micron range has placed increasing demands on the gas supply instrumentation to reduce particle generation. Particle generation can be reduced by improving the surface finish (Ra) and morphology of the wetted internal surfaces of the MFC. Table 1 shows the surface finish of four internal surfaces inside an MFC body which has been machined with *traditional* machining practices.

Surface finish measurements are very useful for detecting possible surface flaws and imperfections, however, it is also necessary to view the surfaces under high magnification. This allows analysis of specific surface flaws in order that the cause and possible solution to the defect can be found.

The following photographs (Figures 2 through 5) were taken using a Scanning Electron Microscope (SEM) at 1000X and 3000X magnification. These photographs represent surfaces 1 and 2 in the MFC body which were machined with *traditional* machining practices.

Figures 2 through 5 show numerous surface defects caused by the machine tools while cutting the metal. These surfaces contain deep fissures which can trap particles and have areas of increased brittleness which could break off and release particles.

Table 1 - Surface Finish of a Traditionally Machined MFC Body

Surface Finish (μ inch) Ra	Surface			
	1	2	3	4
	32-63	63-125	32-63	32-63

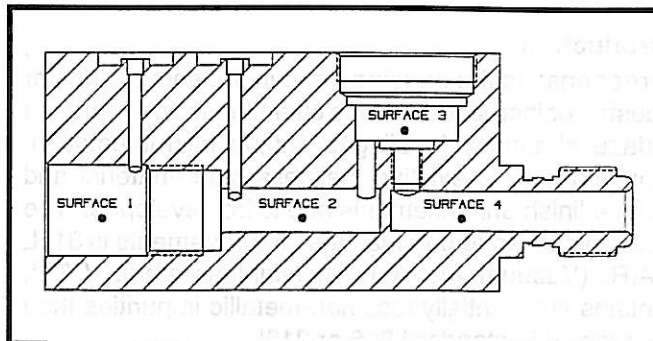


Figure 1 M.F.C. Body Section View

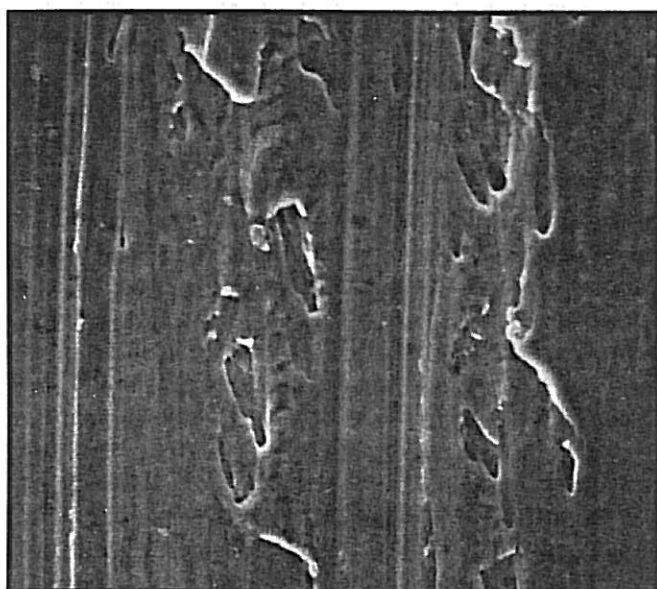


Figure 2 Surface 1 — 1000 Magnification

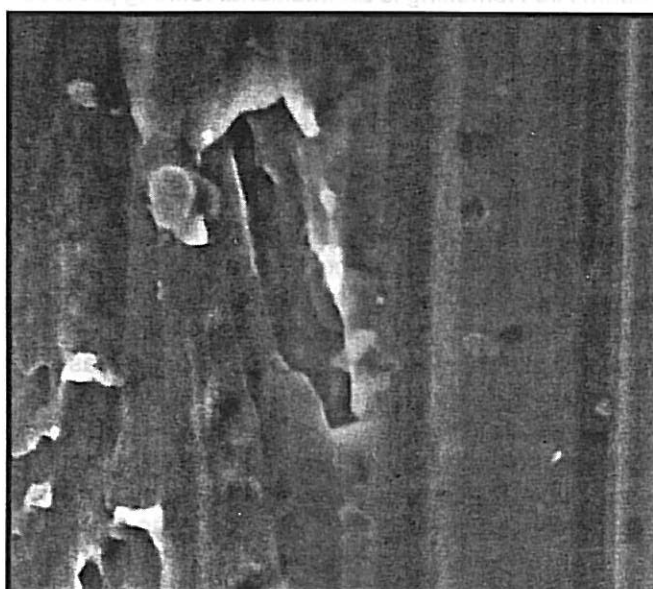


Figure 4 Surface 1 — 3000 Magnification

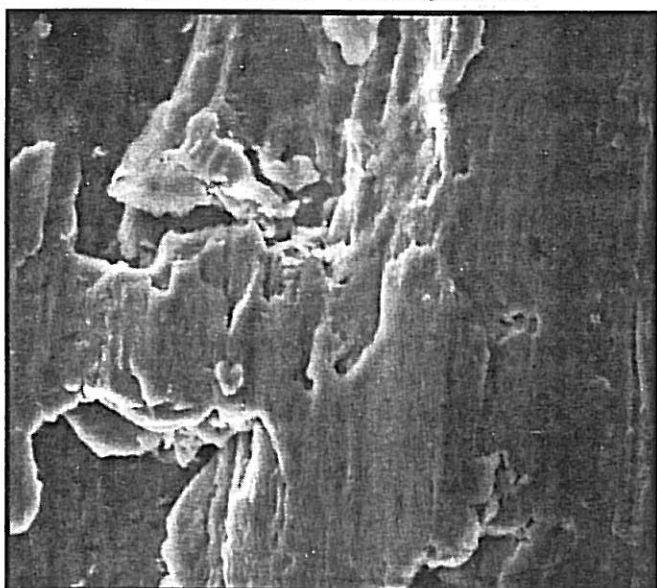


Figure 3 Surface 2 — 1000 Magnification

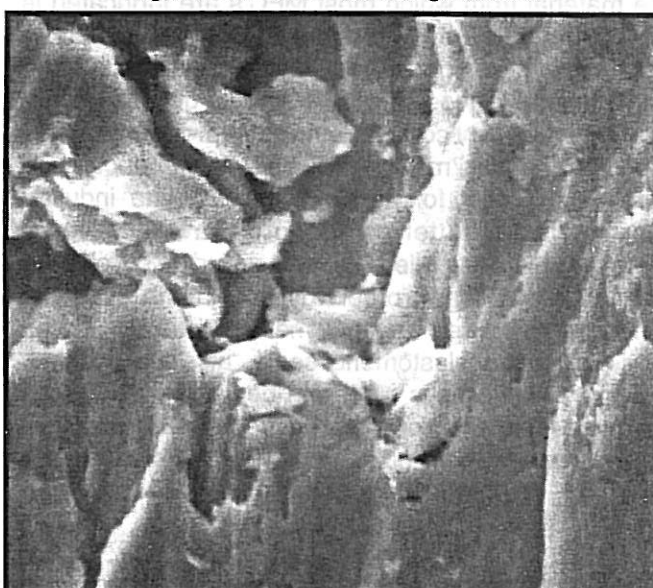


Figure 5 Surface 2 — 3000 Magnification

NOTE: Figures 2 through 5 — SEM photographs of traditionally machined mass flow controller body.

As Figures 2 through 5 reveal, the surface structure of traditionally machined stainless steel offers much room for improvement. To improve these surfaces, several new processing techniques have been employed.

The enhanced processing starts with the initial machining of the internal holes. Tooling, machining process parameters and the type of machining equipment are properly selected to yield the best possible machined surface.¹ After machining, the internal surfaces are subjected to a process called abrasive flow machining (AFM). This process involves extruding a polymeric slurry, which contains cutting abrasives, through the MFC body. This process performs two main functions. First, it removes burrs from all cross-drilled holes and, after removing the burrs, rounds the sharp edges. Second, it improves the surface finish in the smaller diameter holes which

subsequent processing cannot handle. The next process, called "mechanical micropolishing" is used to bring the finish of the largest internal surface area down into the 4-10 μ inch Ra range. Significant attention was given to all processes to ensure that each involved the cutting of metal and not the burnishing or rolling over of metal which can lead to the creation of voids. After the micropolishing process, the parts undergo an electropolishing process. This process will further improve the surface finish as well as improving the surface chemistry of the metal.

Table 2 - Surface Finish of an "Enhanced Process" MFC Body

Surface Finish (μ inch Ra)	Surface			
	1	2	3	4
	4-10	4-10	4-10	4-10

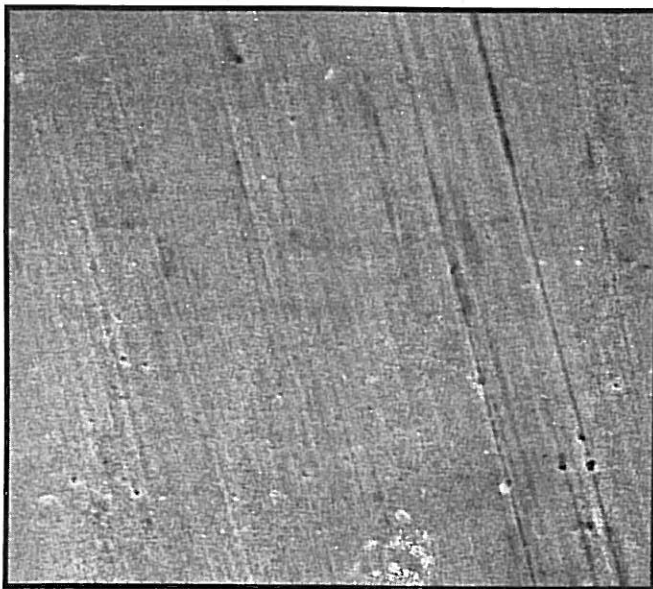


Figure 6 Surface 1 — 1000 Magnification

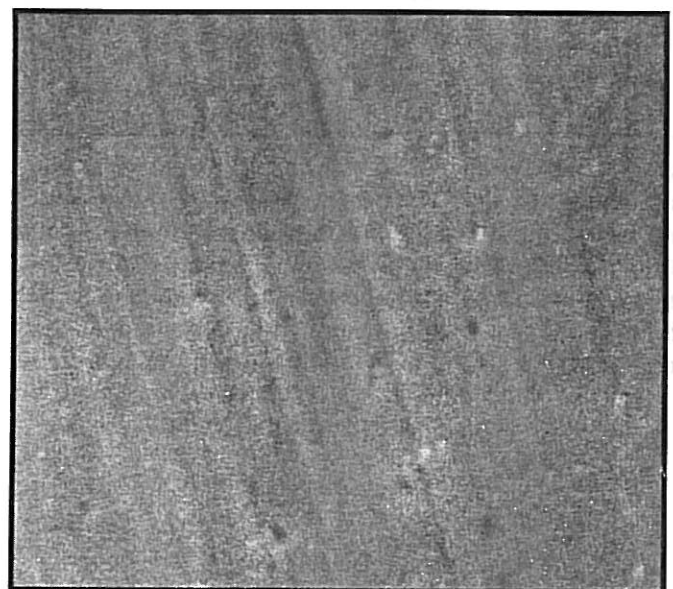


Figure 8 Surface 1 — 3000 Magnification

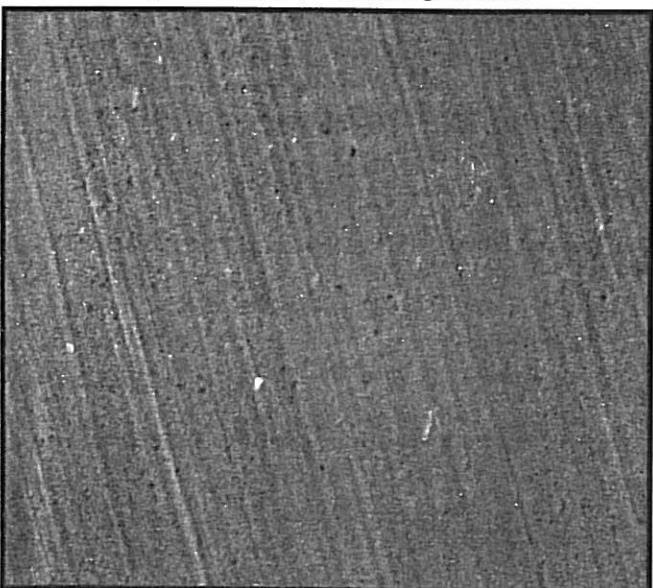


Figure 7 Surface 2 — 1000 Magnification

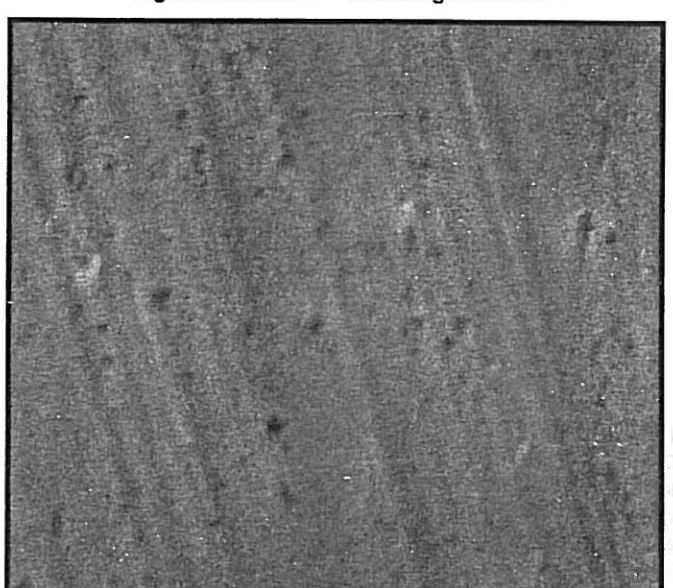


Figure 9 Surface 2 — 3000 Magnification

NOTE: Figures 6 through 9 — Photo micrographs of Enhanced Process MFC body. Brooks 5964 MFC used for SEM.

Table 2 on the previous page shows the surface finish of four surfaces inside an MFC body which has the fully "Enhanced Process." This includes all the previously mentioned processes.

Table 3 shows how the improved surface chemistry of electropolished 316L will reduce its rate of corrosion when compared to 316 or 316L stainless steel.

Table 3 Corrosion Rates of Type 316 Stainless Steel in Hydrogen Chloride²

Coupon Material	Corrosion Rate Mills per Year
316	4.7
316L	2.1
316L (Electropolished)	1.5
Test Gas: Hydrogen Chloride 1500 ppm moisture	
Conditions: Pressure 3-5 psig. Temperature ambient Flow Rate 100 cc/min Duration 200 hours	

When comparing Table 1 with Table 2, a dramatic improvement in surface finish can be seen. The photographs in Figures 6 through 9 were taken with a SEM at 1000X and 3000X magnification. These SEM photographs should be compared with the *traditionally* machined surfaces to appreciate the improvements the additional processing techniques have made.

These pictures reveal smoother, more homogenous surfaces which are unlike the traditionally machined surfaces that had undesirable characteristics such as particle shedding, voids and brittleness.

Conclusion

The purpose of this paper is to give a brief explanation of how recent developments have greatly improved the surface finish and surface morphology of the internal areas of a mass flow controller body. These improvements include the use of new, cleaner, highly corrosion-resistant stainless steel called 316L V.A.R. and new surface processing techniques exclusive to Brooks Instrument MFC products. Dramatic improvements in the surface Ra and overall surface finish have been achieved. Examination under high magnification with a scanning electron microscope verifies these improvements.

Trademarks

Brooks Brooks Instrument, Division of Emerson Electric Co.
Rosemount Rosemount, Inc.

Footnotes

¹ G. Bourscheid, H. Bertholdt, "How Production Technologies Influence Surface Quality of Ultraclean Gas-Supply Equipment", *Microcontamination Magazine* (Feb-May 1990)

² Hardwick et. al., "Ultraclean Processing Environments", *Proceedings of Semicon East, Boston SEMI, Mountain View, CA, 1989*

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