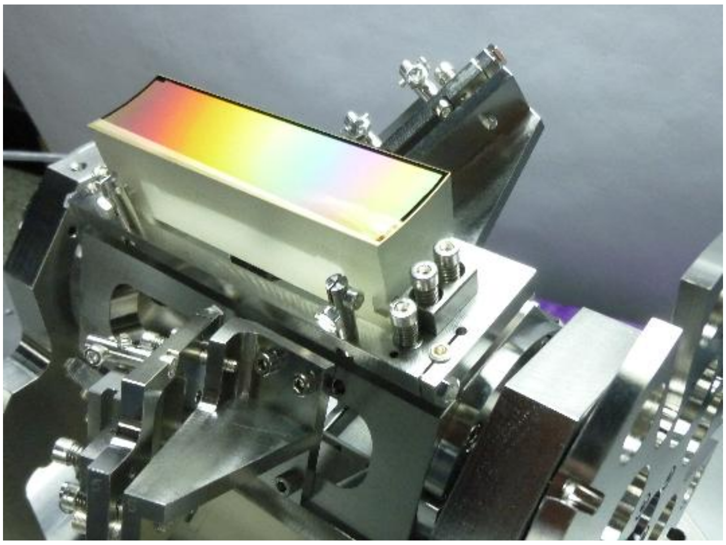
3750g/mm VLS Grating
FERMI at Elettra – Trieste

HORIBA Technical Proposal



Horiba reference: **VUV/180118/CG/386**
Date: **January 18th, 2018**

Index Table

Subject

This document presents the HORIBA Scientific technical proposal for the provision of a plane holographic VLS diffraction grating for FERMI at Elettra -Trieste.

1 General Overview

1.1 HORIBA Scientific (formerly HORIBA Jobin-Yvon)

HORIBA Scientific is part of the Horiba Group which employs over 6900 people worldwide and has a sales level in excess of approximately 1500 million dollars. We are one of the top five companies in the world for optical and analytical instrumentation.

HORIBA Scientific is a state-of-the-art manufacturer of complete VUV beamlines, synchrotron beamline monochromators and components such as chambers, cooled slits, mirrors and gratings.

For over a century, HORIBA has been in the forefront of the development of high quality optical components and spectroscopic systems for chemists, physicists, biologists, engineers and scientists. HORIBA is committed to the development of high quality, user friendly products that make complicated measurements routine and easy.

Founded in 1819, Jobin Yvon has defined the leading edge of optics for spectroscopy. Our leadership in optics has been assured by the continuing development of both ruled and holographic grating technology. This led to the introduction in 1967 of the aberration corrected holographic grating and subsequently ion etched blazed concave and plane holographic gratings.

As a result HORIBA (formerly Jobin Yvon) is able to offer dramatically superior gratings addressing the following technologies:

- Flat field gratings for array detectors.
- Spectroscopy
- Ion etched gratings for the VUV
- Holographic VLS gratings
- Molecular and Dye laser gratings
- Gratings for laser chirped pulse compression, etc…

For synchrotron radiation instrumentation, HORIBA (formerly Jobin Yvon) has always produced instrumentation of high quality and performance and continues to cooperate with the scientific community to bring new products to the marketplace.

HORIBA Scientific has been involved in VUV instrumentation for synchrotron radiation for more than 25 years and has designed and manufactured challenging monochromators and complete VUV beamline systems. Some key instruments are described below:

- In 1975, Jobin Yvon designed and manufactured the first Toroidal Grating Monochromator (patented) in collaboration with LURE (Orsay – France)
- In 1992, Jobin Yvon installed a beamline with variable deviation angle monochromator (Padmore type) at Elettra – Trieste – Italy.
- In 1995, Jobin Yvon designed and manufactured SGM (Dragon) and SM-PGM beamline configurations specifically adapted for third generation synchrotrons.

New generations of ultra-high brightness soft X-ray sources requires more sophisticated instrumentation (since 1980, the brightness has increased by four to five orders of magnitude).

These dramatic improvements have resulted in three main developments at HORIBA:
- new gratings with better efficiency and higher order attenuation,
- improved fabrication techniques for low slope error optical components, and
- the construction of new types of VUV monochromators.

To succeed, HORIBA continues to invest in new ion-etching equipment, coating and polishing facilities, and ray tracing expertise. It has strengthened its collaboration with SOLEIL (France) to easily test new products at their facility.

We have been selected as the main supplier for VUV beamlines and components in several different synchrotron centers (see 1.2. for reference list):

- Super ACO – Lure – France
- BNL – Brookhaven – USA
- ELETTRA – Trieste – Italy
- INDORE – India
- ESRF – Grenoble – France
- NSRL – Hefei – China
- SRS – Daresbury – England
- CLS – Saskatoon – Canada
- SLS – Villigen – Switzerland
- DLS – Oxford – England
- SOLEIL – Gif-sur-Yvette – France

More information about HORIBA Scientific and the VUV products can be found on our web site (www. horiba.com/scientific).

1.2 References

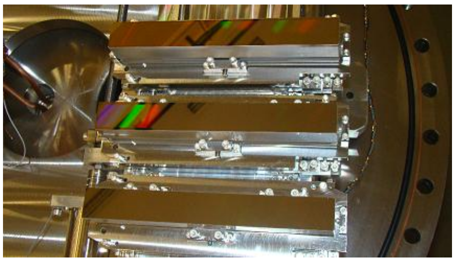
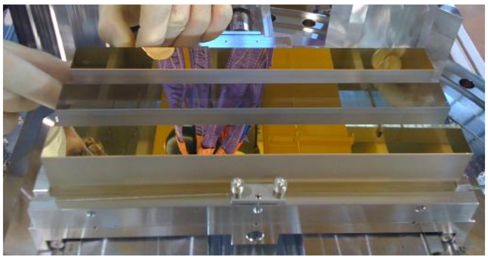




Figure1: FEL metrology grating (left picture) and sets of large size synchrotron gratings manufactured by Horiba (center and right pictures)

The table hereafter presents a non exhaustive list of HORIBA references in the field of VUV optics (mirrors, gratings).

Customer	Country	User	Item
KFA Julich	Germany	Dr Gudat	Toroidal gratings
PTB	Germany	Dr Kuhne	Toroidal gratings
DESY	Germany	Dr Sonntag	Toroidal gratings
BESSY	Germany	Dr Braun	Toroidal gratings
LURE	France	Dr Petroff	Toroidal mirrors Plane gratings
IBM	USA	Dr Eastman	Toroidal gratings
IBM	USA	Dr Himpsel	Toroidal gratings
Philips	Netherlands	Dr Larsen	Toroidal gratings
SSRL	USA		Toroidal gratings
BNL	USA		Toroidal gratings
Frascati	Italy	Dr Piancentini	Toroidal gratings
LURE	France	Dr Wuillemier	Toroidal gratings VUV mirrors
CEA Saclay	France	Dr Nenner	Toroidal gratings VUV mirrors
University of Virginia	USA	Dr Schnatterly	Toroidal gratings
University of Winconsin	USA	Dr Eastman	Toroidal gratings
Los Alamos National Laboratory	USA		Toroidal gratings
University of Tokyo Synchrotron Radiation Lab	Japan		Toroidal gratings
Imperial College London	England	Dr Connerade	Toroidal gratings
University of Hamburg	Germany	Dr Zimmerer	Toroidal gratings
DESY	Germany	Dr Bruhn	Toroidal gratings VUV mirrors
DESY	Germany	Dr Schmidt	Toroidal gratings VUV mirrors
LPSP	France	Dr Delaboudinière	Toroidal gratings

Princeton University	USA		Toroidal gratings
CEA Saclay	France	Dr Lompre	Plane Gratings VUV mirrors
Ecole polytechnique	France	Dr Benattar	Plane Gratings VUV mirrors
ELETTRA	Italy	Dr Perfetti	Spherical gratings SiC
ELETTRA	Italy		Spherical gratings SiC
ELETTRA	Italy	Dr Tondello	Spherical gratings VUV mirrors
Frascati	Italy		Toroidal gratings
LURE	France	Dr Thiry	Plane gratings VUV mirrors
TSUKUBA	Japan		Toroidal gratings
Nuclear Physic Institute Novosibirsk	Russia		Toroidal gratings
Argonne National Lab	USA	Dr Glushkin	Spherical gratings
BNL	USA	Dr Chen	Spherical gratings
SRRC	Taiwan		Spherical gratings
PAL Pohang	South Korea		Spherical gratings
ESRF	France	Dr Goulon	Spherical gratings SiC
Uppsala University	Sweden	Dr Nordgren	Spherical gratings
Trieste	Italy	Dr Braïcowich	Toroidal gratings VUV mirrors
Inter University Consortium Indore	India	Pf Srinivasan	Toroidal gratings
Centre for Advanced technology Indore	India	Dr Nandedkar	Toroidal gratings
Bombay Atomic Research Centre	India	Dr Rao	Toroidal gratings
Lund Institute of Technology	Sweden	Dr Walhlström	Plane gratings Toroidal mirrors
LURE	France	Dr Sirotti	Spherical gratings VUV mirrors
ESRF	France	Dr Susini	Plane VLS gratings
CSIC Madrid	Spain	Dr Asensio	Plane VLS gratings VUV mirrors
LURE	France	Dr Nahon	Spherical gratings VUV mirrors
Tsukuba	Japan	Dr Ito	VLS gratings
ELETTRA	Italy		VLS gratings
Tokyo University	Japan	Dr Fujisawa	VLS gratings
Hefei	China		VUV gratings VUV mirrors
Daresbury	England	Dr Quinn	Toroidal mirrors
BESSY II	Germany	Dr Senf	Spherical gratings VUV mirrors
Daresbury	England	Dr Shaw	Toroidal gratings
ELETTRA	Italy	Dr Masciovecchio	Spherical mirrors
BESSY II	Germany	Dr Reichard	Plane gratings
BNL	USA	Dr Hulbert	Spherical gratings
Riken	Japan	Dr Watanabee	Spherical gratings
University Of Tokyo	Japan	Dr Mizokawa	Spherical gratings
Diamond	England	Dr Dhesi	Toroidal Mirrors
SLS	Switzerland	Dr Johnson	Plane Grating Unit
SOLEIL	France	Dr Polack	VGD VLS gratings

1.3 Optics Manufacturing and tests

The HORIBA Scientific laboratories and production facilities are located at Longjumeau, a suburb of Paris. The Longjumeau plant houses the largest and most comprehensive and complete grating facility in the world.

Figure 2: HORIBA new facilities

Glass blanks for gratings are cut and polished to a surface flatness better than "$\lambda/10$" in our own facility. The glass is carefully selected from many different kinds of materials and only the finest glass is used. After extensive machining and hand polishing procedures, the glass is subjected to critical testing and, if approved, forwarded to one of the grating labs.

For high flux synchrotron radiation sources, HORIBA was the first to produce high quality gratings on exotic blank material, such as silicon carbide and silicon on either toroidal or spherical blanks.

In order to achieve the ultimate in recording a holographic grating, our holographic grating labs are contained in underground rooms. Great attention has been paid to eliminate problems concerning temperature, vibration and reliability of operating conditions.

Lasers, collimators, optical components, developing and processing equipment are all available.

Coating and chemical operations are also performed in our own processing laboratories. The labs are geared to accommodate all the company's replication and deposition requirements with equipment such as fully computerized high vacuum evaporation systems.

All of the equipment involved in the handling and processing of gratings and blanks is enclosed in clean rooms with laminar flow systems, which provides a class 100-dust free environment.

In order to assure each customer of the quality and precision of the grating he purchases, HORIBA Scientific assigns a serial number to each grating.

The specifications and test results of the grating are then registered and given to the customer in form of a "Specification Registered Certificate".

This represents our personal guarantee to the customer as to the characteristics of his particular grating.

ISO 9001 and **ISO14001** certified HORIBA warranties the quality and the consistency of its production.

HJY production and test facilities access is strictly limited to authorized HJY production team.

1.3.1 Optic Blanks

HORIBA Scientific has developed full capabilities for polishing of optic blanks following custom specifications. Possible shapes are plane, spherical, cylindrical or toroidal. Material can be Pyrex, ULE, Fused Silica, and Silicon. **HORIBA Scientific can provide very large optics up to meter size scale.**

Typically, HORIBA Scientific offers a polished surface quality with the following specifications (depending on the optic substrate):

- Surface micro-roughness: 0.2 to 0.5 nm RMS typical (plane or spherical blank)
  Slope error (on useful area): up to ≤ 0.1µrad RMS

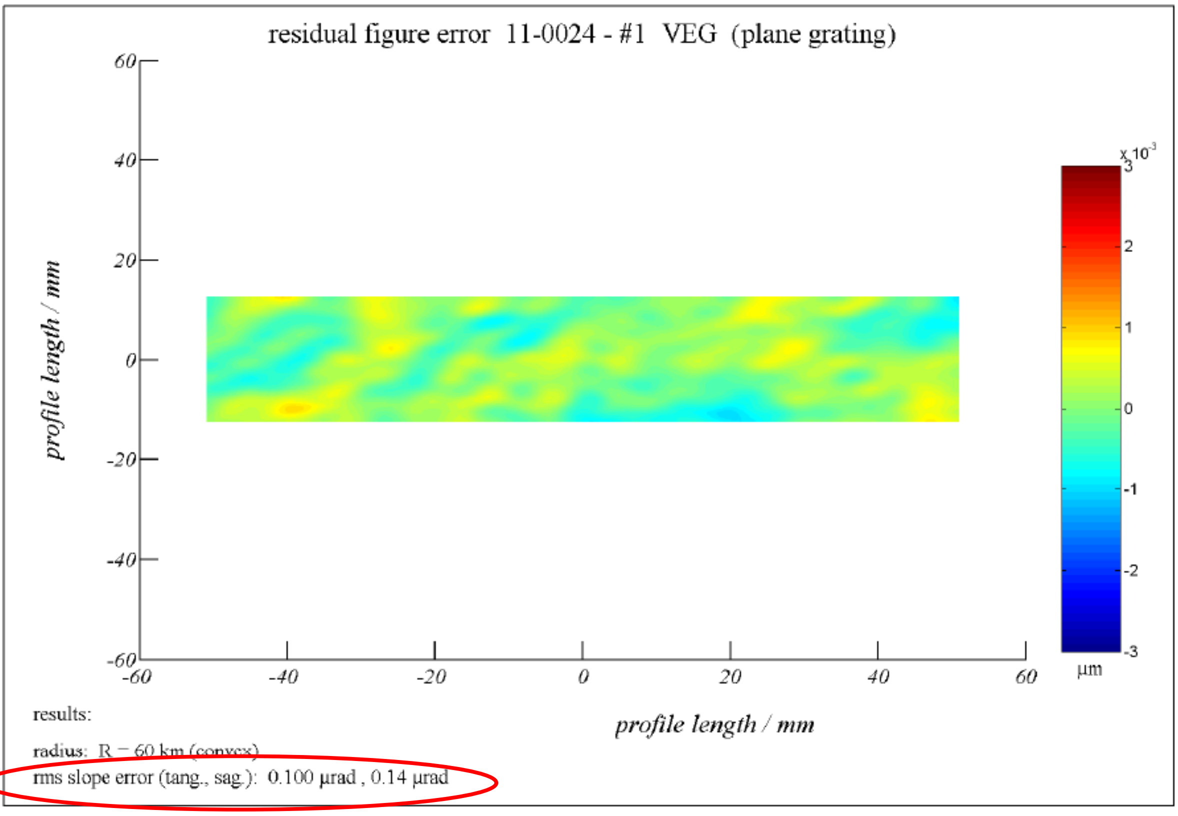


Figure 3: Example of slope error measurement ≤ 0.1µrad RMS of synchrotron substrate

1.3.2 Variable Line Spaced Holographic Diffraction Grating

Highly polished and figured blanks are coated with a layer of photosensitive material and then exposed to interference fringes created at the interference of two coherent laser beams. Chemical treatment of the photosensitive layer selectively dissolves the photoresist layer forming grooves in relief.

The geometry of the two laser wavefronts used to produce the interference fringes is calculated to produce a non-uniform groove distribution (VLS law) on the surface according to the requirement of the user. In Section 3, we present the result of the optical calculation we carried out to define the most suitable holographic recording setup for your grating.

Depending on required VLS law it is possible or not to use two spherical wavefronts to record the grating.
The geometry of the recording optical setup is obtained thanks to proprietary software developed by HORIBA Scientific grating R&D team. This software determines the most suitable holographic recording configuration that enables the recording of the holographic VLS grating.

After holographic recording manufacturing step, the photosensitive layer is submitted to a chemical treatment, which permits to selectively dissolve the photo resist layer. Following this step a pseudo sinusoidal groove shape appears on the grating surface.

1.3.3 Diffraction efficiency and optimization

1.3.3.1 Groove to Period Ratio Definitions

Groove profiles of Ion Etched Laminar Gratings present a trapezoidal shape.
The trapezoidal shape is defined by the Grating Constant *d*, the Groove Height *h*, the groove width *c* and the Angle *α* between the edges and the base.

Groove to Period Ratio can be defined in several ways:

One way is to consider Groove Width measured on top of the profile: Top Groove definition. In this case, the groove width includes the edges of the trapezoid.

Another way is to consider Groove Width measured at mid height of the profile. This 2nd way is the way that we use currently at HORIBA Scientific.

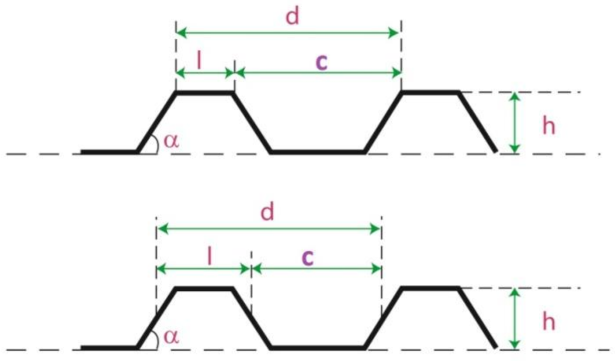


Figure 4: Groove to period ratio scheme

The length of the projection of the edge is ($h/\tan\alpha$).

If Groove Width defined at top is called *c* (Top) and Groove Width defined at Mid Height is called
c (Mid H) we have :

c (Top) = c (Mid H) + ($h/\tan\alpha$)

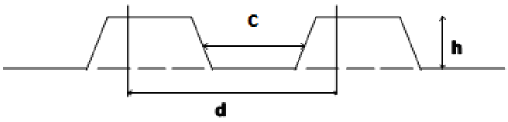
Profile at Mid Height the relation between the 2 definitions is:

$$c/d \text{ (Mid H)} = c/d \text{ (Top)} - (h/\tan\alpha)/d$$

1.3.3.2 Optimization of diffraction efficiency

Spectral efficiency depends on the shape of the groove profile, which is defined by:

- The depth *h*
- The valley half-height width *c*
- The groove spacing *d*



For a given configuration (diffraction angle, incidence angle), the ratio *h/d* and *c/d* are determined to optimize the first diffraction order spectral efficiency and/or minimize the contribution of the high harmonics.
To optimize the groove shape parameters *h* and *c*, we can perform calculation based on the Electromagnetic Diffraction Theory with proprietary software developed in collaboration with SOLEIL (France). To improve and validate this calculation, experimental measurements are continuously performed with SOLEIL optical group. The measurements already made shown a good agreement with the calculation.

1.3.4 <u>Ion-etched gratings with laminar groove profile</u>

The groove laminar shape manufacturing process includes three steps:

1. Recording and adequate chemical treatment of a quasi-laminar (trapezoidal shape) holographic grating defined by the parameters h_m and c_m. h_m and c_m are pre-determined according to the expected final goal.
2. Optimization (h_{goal} and c_{goal}) and transformation of the quasi-laminar shape in the laminar shape in the substrate by ion-etching process. The previously manufactured grating is used as a mask. Side walls of the groove are transferred in the substrate with modification of intercepting angles.
3. Removal of the remaining photoresist layer.

The final grating is wholly engraved into the substrate (baking out procedure is allowed).

Ion-etching process is particularly well suited for monitoring the fine sculpting of the shape.

Because the depth of VUV gratings is very small, ion-etching process does not affect the micro-roughness of the fine structure of the high polished substrate. Others properties, like straylight, are preserved.

An Atomic Force Microscope is available in HORIBA Scientific control laboratory to test grating groove profile. A typical example of such AFM profile is shown Figure 1.

Uniformity of the sculpting along the whole surface is ensured by a careful adjustment of the interferogram pattern and by a careful control of ion beam section and process optimization.

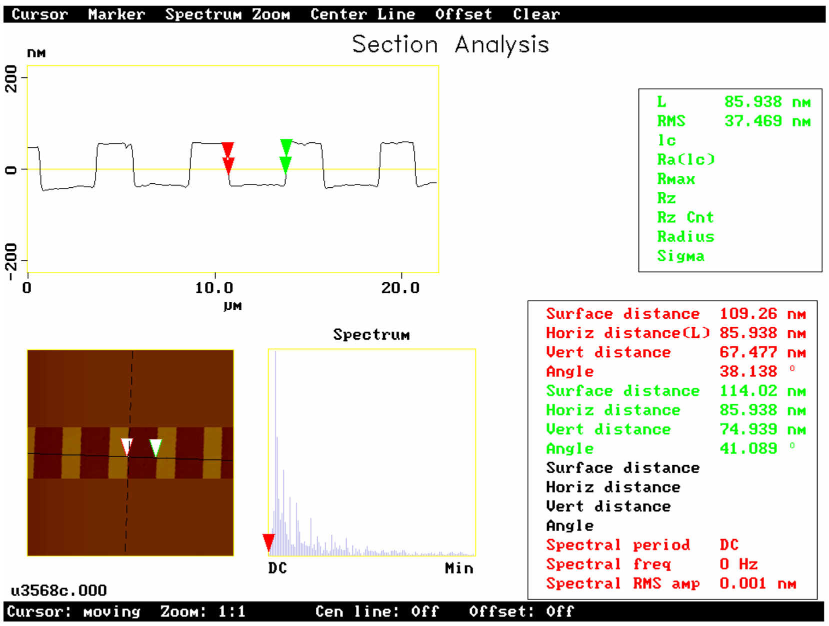


Figure 5: Groove profile measurements; Atomic Force Microscope observation

1.3.5 Reflective coating

Metallic coating is deposited under cryogenic vacuum. The thickness is monitored thanks to quartz controller.
Complete grating processing is done in cleanroom, grating is handled only with gloves in order to avoid carbon contamination.

1.3.6 Test methods

1.3.6.1 Blank surface

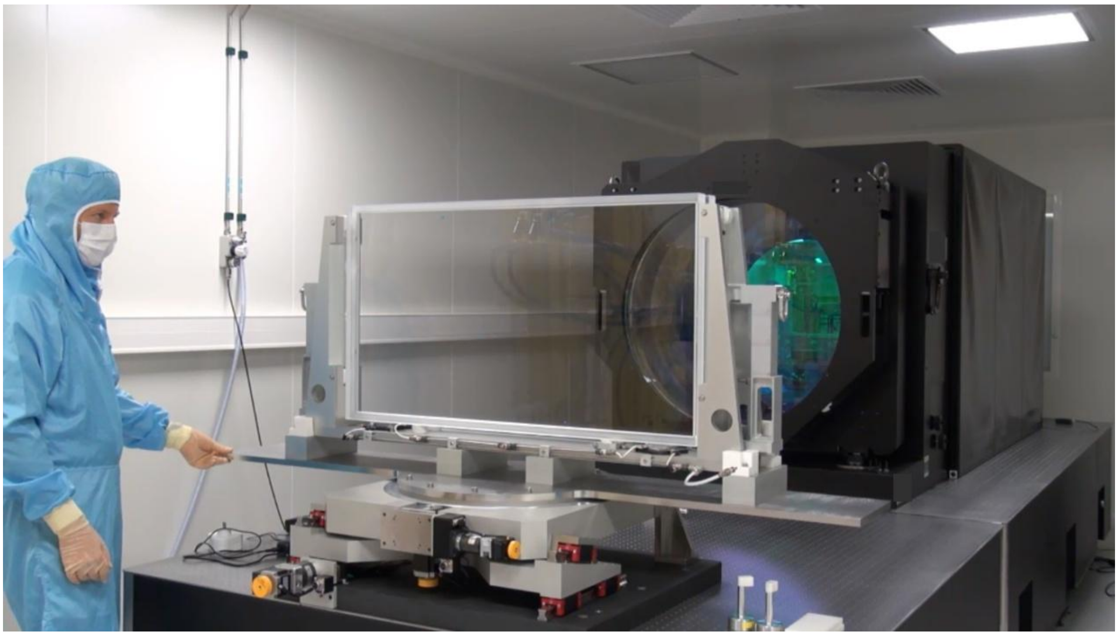


Figure 6: HORIBA large scale interferometer setup

Micro-roughness RMS is tested with a Nomarski interferential microscope (magnification up to x 200) over the whole surface by comparison with reference samples during blank polishing in our polishing laboratory.
After polishing, quantitative measurements are performed using a Micromap installed in HORIBA Scientific control laboratory.

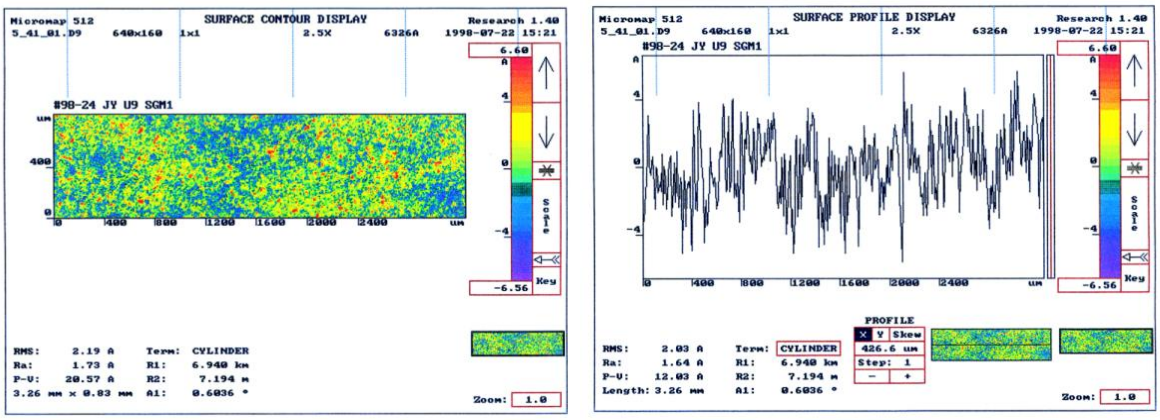


Figure 7: Examples of microroughness quantitative measurements. Micromap Observation

Slope-error is tested with Wyko interferometer in HORIBA Scientific control laboratory during polishing process. Correlation of slope error RMS measurement with Long Trace Profiler (LTP) of the ESRF (Grenoble/France), Brookhaven National Lab (USA), ELETTRA (Trieste/Italy) and SOLEIL (Gif sur Yvette/France) Metrology Laboratory have shown very good agreement.

Radiuses of curvature of the mirror are controlled during polishing process thanks to calibrated samples measured by an independent national laboratory the “Institut d’Optique” (Palaiseau/France).

1.3.6.2 Visual inspection

The optical pieces are submitted to visual inspection under grazing incidence with visible illumination and micro-structure is checked with Nomarski interferential microscope.

1.3.6.3 Diffraction efficiency

Groove shape parameters are tested with the Atomic Force Microscope (AFM).

1.3.6.4 Test documentation

A complete report including all measured data will be established for each optical piece. Test documents will be delivered together with the optical pieces.

1.4 Cleanliness

During the whole manufacturing and testing program, our facilities, processes and procedures ensure the highest standard of quality necessary for ultra-high vacuum operation.

1.5 Packaging

The diffraction gratings, including packaging operations as well as during the whole manufacturing and testing program ensure the highest standard of quality in order to be thoroughly clean and free from any organic low vapor pressure material or organic fluid.

1.6 Packing and Delivery

Each optic will be packaged in its own protective container. The container is in massive Teflon, and is designed to prevent dust, moisture, contamination of the optical piece and prevent contact of any object with any part of the clear aperture.
The packaging of each optic will clearly identify its serial number.
Packing for shipment will insure that each optic is insulated from severe shock and rough handling. Each optic will be delivered with its own documents: optical test report and conformance certificate.

2 Technical proposal

2.1 Technical specifications of 3750 l/mm grating

Horiba Scientific proposes the following gratings specifications. The grating is holographically recorded with the given VLS law and ion etched inside the silicon substrate.
The table below indicates the Horiba specifications related to customer requested items for which we can commit, for all other requirements not mentioned in the specifications list below, we do not specify them.

ITEM	REQUIREMENTS	HJY SPECIFICATIONS	CONTROL METHOD
Blank material	Fused Silica	Fused Silica	Supplier certificate
Blank dimensions	Width = 25 mm Length = 250 mm Thickness = 40 mm	Width = 25 mm Length = 250 mm Thickness = 40 mm	Dimensional measurement
Blank Useful area	W20 x L240 mm²	W20 x L230 mm² (W20 x L240 mm² on a best effort basis)	Dimensional measurement
Blank shape	Plane with |R|>30 Km in both directions	Plane with |R|>30 Km in both directions	-
Blank slope errors	Sagittal : ≤ 0.5 µrad RMS ; Tangential : ≤ 5 µrad RMS ;	Sagittal : ≤ 0.7 µrad RMS (≤0.5µrad RMS best effort); Tangential : ≤ 5 µrad RMS ;	Interferometry
Blank micro-roughness	≤ 0.4 nm RMS ;	**≤ 0.3 nm** RMS ;	Interferometry
Grating ruled area	20mm Wide (W) 80 mm Long (L) located in the central part of the substrate	24mm Wide (W) 84 mm Long (L) located in the central part of the substrate with 2mm border (not specified) all around the ruled area.	Dimensional measurement
Grating useful ruled area	20mm Wide (W) 80 mm Long (L) located in the central part of the substrate.	20mm Wide (W) 80 mm Long (L) located in the central part of the substrate.	Dimensional measurement

Grating Line density	**VLS law :** N=N0*+N1*X+N2*X^2+... Where N0 is groove density in lines/mm (X is in mm) at the grating center for X = 0 mm. N0 = **3750** ± 0.5% mm^{-1} N1 = **2.64757** ± 1% mm^{-2} N2 = **0.0013367** ± 2% mm^{-3} N3 = **$5{,}0.10^{-10}$** ± 10% mm^{-4}	**VLS law :** N=N0*+N1*X+N2*X^2+... Where N0 is groove density in lines/mm (X is in mm) at the grating center for X = 0 mm. N0 = **3750,01**mm^{-1} +/- 0.03 % mm^{-1} N1 = **2.65** +/- 1.7 % mm^{-2} N2 = **0.0013** ± +/- 19 % mm^{-3} N3 = **$2.8.10^{-6}$** +/- 34 % mm^{-4}	Manufacturing process control*
Energy range	1.05 - 9.6 nm	1.05 - 9.6 nm	-
Constant incident angle	88 deg	88 deg	-
Groove shape	**Lamellar Groove Shape with:** Groove depth : h = 3 - 5 nm Groove ratio : c/d = 0.6 - 0.8	**Lamellar Optimized Groove Shape with :** Groove depth : h = 4 nm ± 1.5nm Groove width/spacing ratio : c/d = 0.7 ± 0.1	AFM
Coating	Gold (50 nm) (no Cr binding layer allowed)	Au 40nm ± 10nm (no Cr layer)	Quartz monitoring

**: The VLS law is managed during the manufacturing process. VLS law is guaranteed by the geometrical setup, optics and laser wavelength.*

We will measure the groove profile and the grooves micro-roughness with AFM. We do not measure the diffraction efficiency. The efficiency can be calculated from the measured AFM groove shape.

2.2 Manufacturing tolerances of 3750 l/mm grating VLS law

N = N0 + A1*Y + A2*Y^2+..
Where N0 is groove density in tr/mm (Y is in m) at the grating center for Y = 0 mm.

Tolerancing of HJY :

		Spec.	HJY	HJY Production Tolerance
Polynomial comparison	N_0 (gr/mm)	**3750**	**3750,01**	**+/- 1,1 (0,03 %)**
	N1 mm^{-2}	**2.64757**	**2,65**	**+/- $4{,}4.10^{-2}$ (1,7 %)**
	N2 mm^{-3}	**0.0013367**	**0,0013**	**+/- $2{,}5.10^{-4}$ (19 %)**
	N3 mm^{-4}	**$5{,}0.10^{-10}$**	**$2{,}8.10^{-6}$**	**+/- $9{,}3.10^{-7}$ (34 %)**

On the following graph we present the difference between the requested VLS Law and the one given by the holographic recording setup in groove density along the useful area of the grating.

Graph of the dN error along x for the HJY nominal grating :

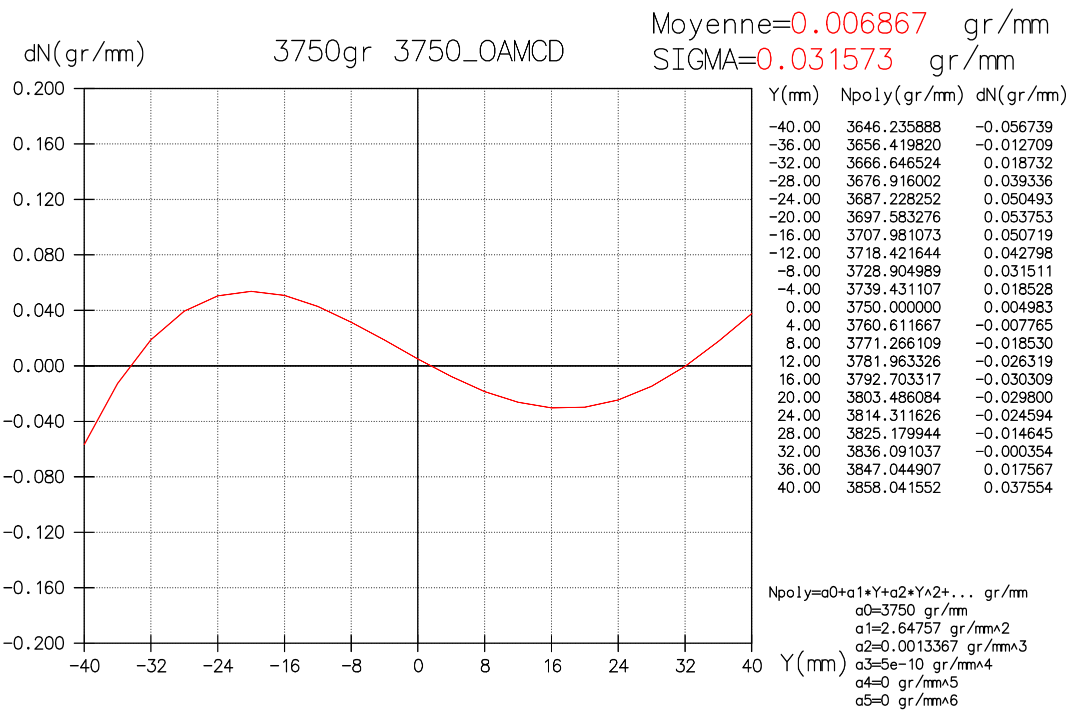


Figure 8: Plot of the difference ΔN between the polynomial law recorded thanks to the holographic process and the exact VLS groove density versus the coordinates at the grating surface

The following graph shows the curvature of the groove, with a magnification of 200000, produced by the holographic method. It is obvious from this graph that this effect will be negligible compare to all other sources of error.

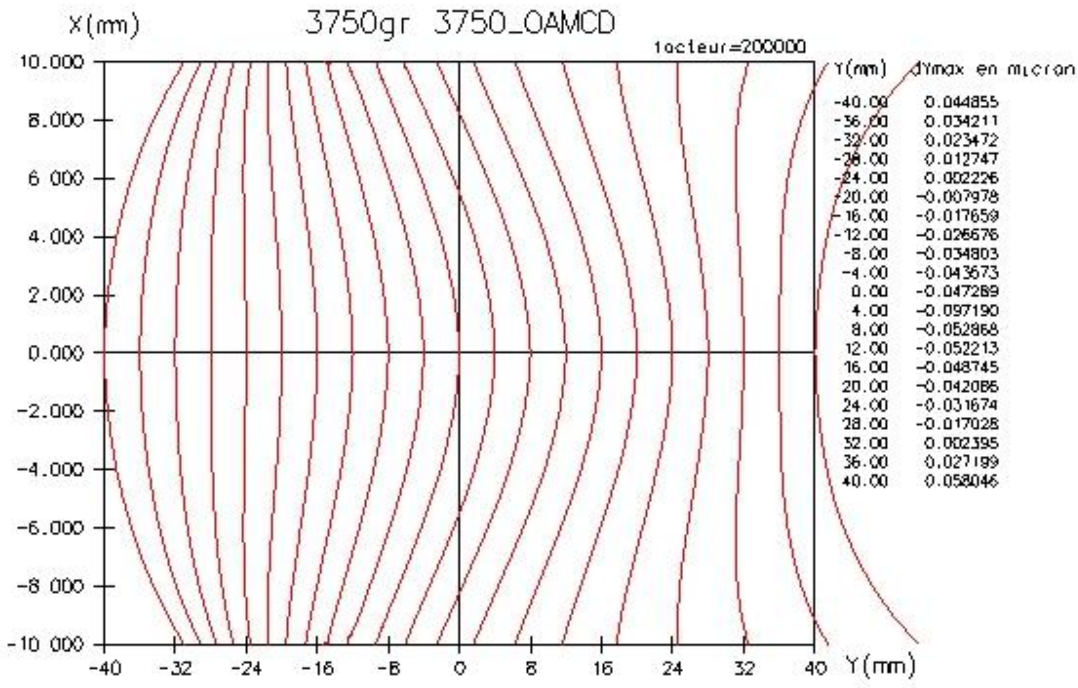


Figure 9: Plot of the groove shape along the grating height (Factor = 200000): the deviation in Y direction from a straight line is scale d by a factor 200000.

3 Mechanical drawing

3.1 3750 l/mm grating

To be confirmed by customer at the time of order.

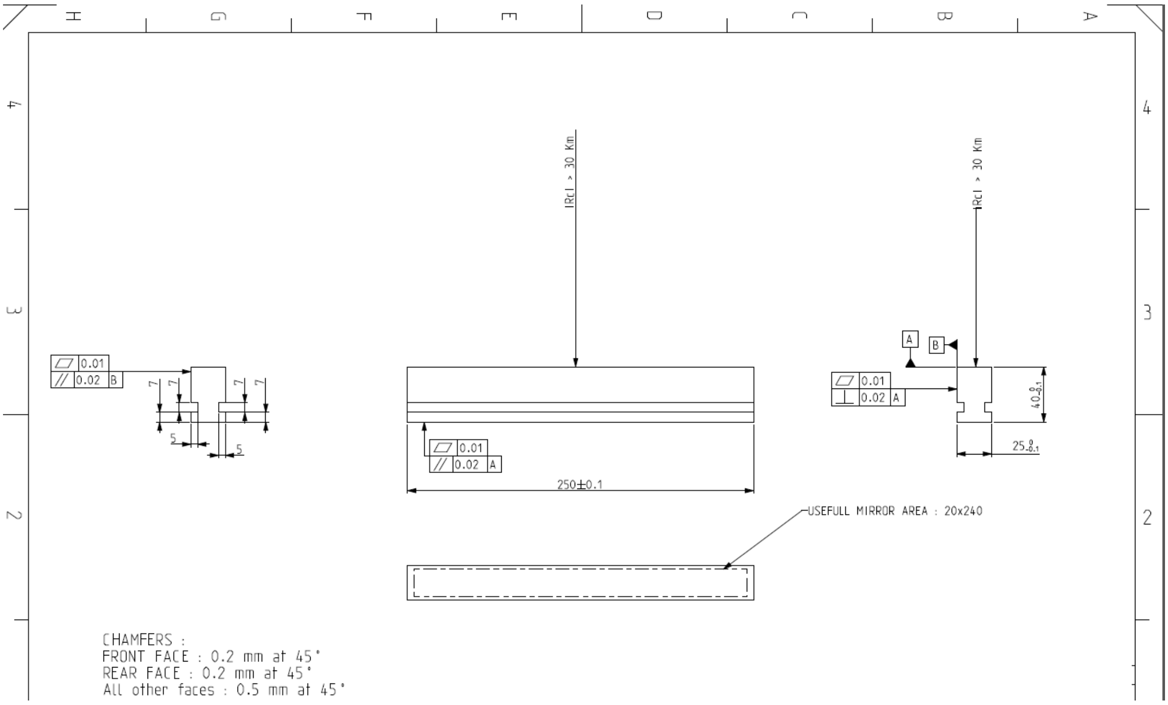


Figure 10: Mechanical drawing of the 3750 l/mm grating fused silica substrate

4 Ion etched, holographic gratings manufacturing quality flow chart

Note: Step presented in orange outlines critical point for delivery

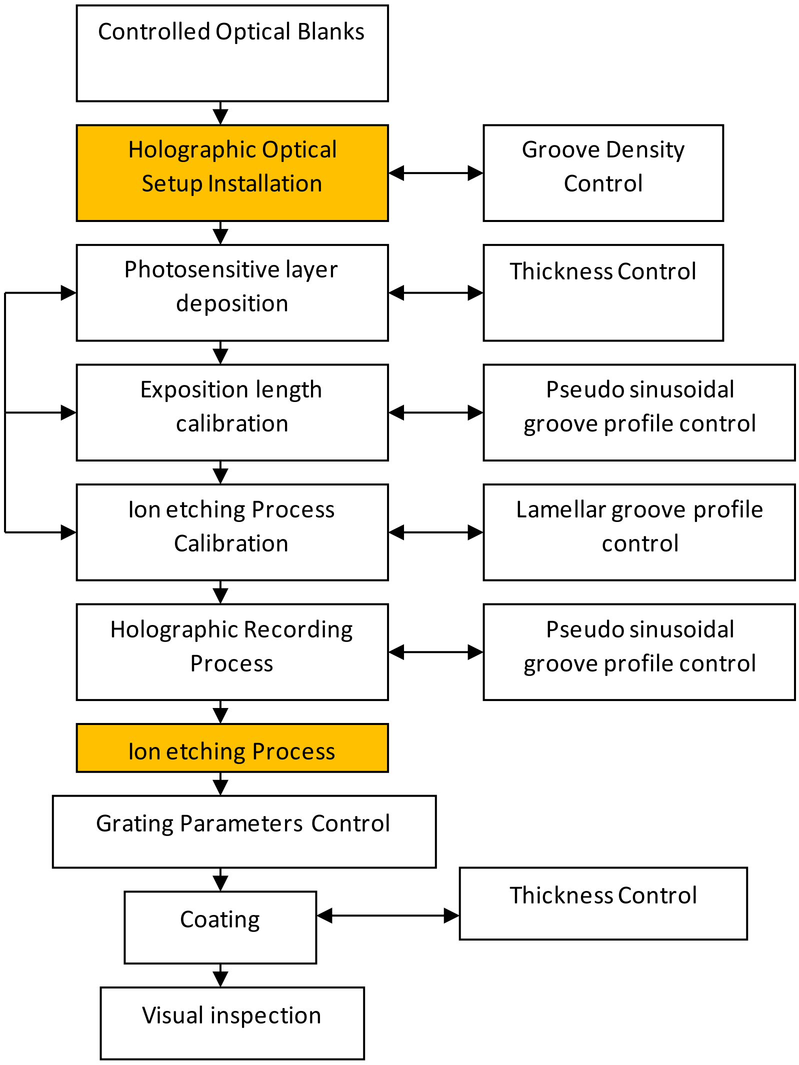


Figure 11: Ion-etched grating manufacturing flow chart

5 Gratings handling guidelines

Optical gratings can be easily damaged by moisture, fingerprints, aerosols, or the slightest contact with any abrasive material.
Gratings should only be handled when necessary and always held by the sides. Contacts to the front ruled surface may permanently damage this surface.

The following precautions apply:

- Wear a facemask when working with the grating, to avoid contaminating the surface.
- Latex gloves or a similar protective covering should be worn to prevent oil from fingers from reaching the grating surface.
- Do not touch the surface of the grating with anything. There should be no mechanical contact with the grating grooves.

No attempt should be made to clean a grating. Any attempt to clean a grating with a solvent voids the warranty. If there are some dust particles on the grating, flush with inert gas (pure dry nitrogen). **It is critical that the gas must be very clean. <u>Do not use air cans</u>. The CFC-like pressure gas may react with the metal reflective coating on the grating surface when exposed to light radiation. Watch out for compressed air as most compressors add to the air some oil residues that can contaminate the gratings.**
Scratches or other minor cosmetic imperfections on the surface of a grating do not usually affect performance.
For transportation or storage, always use the initial packaging: For Teflon containers, the grating must be put ruled surface down, back surface up. The grating must be maintained by the screws to avoid vibrations during transportation. For plastic covers, always use Kapton® tape to maintain the protective cover in contact with the ruled surface then seal inside a plastic or aluminium bag to protect the grating from any outgassing residue from the packaging foam and /or the surrounding environment.

6 Acceptance test report

HORIBA provides with each grating a full Acceptance Test Report (ATR) with following measurements:

- Dimensional mechanical controls,
- Groove density measurement
- Groove profile measurement,
- Efficiency recalculation from groove profile AFM measurement
- Slope error measurement,
- Micro-roughness measurement
- Error on VLS terms k0, k1, k2
- Detailed map of cosmetic defects.

Our diffraction gratings are compliant to REACH and RoHS legislations.