

**NMS Programme for Length
(1999 – 2002) MPU 8/61**

**Review of methods for
cleaning surfaces prior to
surface texture measurement**

Project LE9953, milestone 5.3.1.4

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September 2001

Review of methods for cleaning surfaces prior to surface texture measurement as part of project LE9953, milestone 5.3.1.4.

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ABSTRACT

Meaningful measurements of surface texture can only be made on surfaces free from dirt and contaminants. A literature review of cleaning techniques designed to remove surface contaminants has been conducted. Experimental work to assess the efficacy of current NPL cleaning processes has also been carried out. The alternative methods of cleaning offered no significant advantage over existing NPL techniques. The NPL cleaning processes were found to be suitable for the preparation of samples for surface texture measurement.

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ISSN 1469-4921

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Approved on behalf of the Managing Director, NPL
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1. INTRODUCTION

The measurement of surfaces and surface parameters is increasingly used for process and quality control in many disciplines including information technology, transport, manufacturing industry, precision optics and biosciences. In machine processing, for example, evaluation and control of surface characteristics may be critical as these characteristics often affect the performance, quality and service life of a product. As a consequence there is increasing demand for reference artefacts that have quantifiable surface characteristics. In order to make meaningful measurements of surface texture it is crucial that the surface should be free from dirt and other contaminants that may affect the measurement results.

A range of distinct requirements have resulted in the development of a range of cleaning methods specific for a given task. Many of these methods involve automated industrial processes and may be unsuitable for scaling down to meet laboratory conditions and safety requirements. This report examines some of the surface cleaning processes currently in use and gives the results of a series of experiments designed to test the efficacy of the existing cleaning procedures as part of the sample preparation when making surface texture measurements. The definition of what constitutes surface cleanliness is relative and varies from user to user making a precise definition of cleanliness virtually impossible. This report has, therefore, adopted the definition proposed by Trumpold and Frenzel (2000) which states that “*a surface is clean when any residual contamination fails to affect the functionality of that surface*”. In the case of surface texture reference artefacts the topography of the surface must remain unaltered after cleaning in order to make surface texture measurements that are repeatable and reproducible.

2. LITERATURE REVIEW

The main purpose of this report was to assess the suitability of the existing cleaning methods used as part of the surface texture research and development and measurement services in the Dimensional and Optical Metrology Team at NPL. However, it was considered sensible to first carry out a review of the literature in order to identify other methods and to consider possible alternatives.

Wet cleaning techniques feature prominently in much of the literature and are the preferred methods for cleaning optical surfaces, particularly in the manufacture of precision coated optical components where surfaces need to be free from dirt and contamination before coating. The basic wet cleaning process, described by Talim (1980), involves the removal of contamination by a suitable solvent, *e.g.* ethanol, and/or a detergent, the subsequent removal of the detergent (if used) using deionised water, so as not to re-contaminate the surface, and finally drying of the surface.

It is important to note that some cleaning systems can physically and/or chemically modify the specimen, for example by etching, oxidation and/or reduction. The choice of cleaning agent is, therefore, an important consideration in the cleaning process

(DeLiso *et al* undated)¹. Grosskopf (undated) recommends the use of chemical additives to detergent solutions to reduce the development of latent scratches on glass surfaces cleaned using ultrasonics. A review of cleaning equipment and processes for cleaning optical glass highlights the use of extremely pure water and an ultrasonic bath as an effective cleaning method (Jarratt 1997).

Anghel and Chetwynd (2000a, b, c, d, e) have thoroughly investigated the use of suitable concentrations of various detergents manufactured by Decon™ (Decon Laboratories Inc) combined with the use of an ultrasonic bath on glass surfaces, electroformed nickel surfaces, polycarbonate surfaces, polyvinyl chloride surfaces and silicon surfaces. They give recommendations for suitable cleaning techniques for each of these materials together with the limitations of the techniques. Further investigations (Anghel and Chetwynd undated) have concluded that there is a wide “safe-zone” of conditions under which glass and nickel surfaces can be cleaned effectively using very simple techniques.

Large scale wet cleaning is used in the production of semiconductors often as part of an automated industrial process (*Semiconductor International* 2000). The cleaning fluids tend to be more abrasive in their action than those used to clean glass. For silicon wafer processing, for example, the standard cleaning fluid is based on hydrogen peroxide mixed with ammonium hydroxide or hydrochloric acid. Schmidt *et al*, (undated) have reported on a measurement tool developed to investigate the stability of hydrogen peroxide in different mixtures with respect to temperature and metal contamination. The efficiency of the cleaning process, particularly with respect to the removal of particles from surfaces, has been shown to be improved by the use of high frequency acoustic power (megasonics) (Gale *et al* 1996, Adkins *et al* 1994, Hall *et al* 1994). More recently a wet cleaning process using pure water and chlorine gas has been developed (Shiramizu and Yoshimi 2001).

The ability to reduce the critical dimensions on silicon wafers has meant that increasingly low levels of contamination may present problems. This has led to the demand for more advanced cleaning methods. Small *et al* (1998) recommend replacing deionised water rinses with a buffered rinse solution to remove residual adsorbed metals. A review of cleaning processes for silicon wafers, together with the use of new alternatives, has been carried out by Singer (1995).

In recent years there has been a demand for more cost effective and environmentally friendly technologies due to environmental concerns and health and safety legislation together with the ever present need to reduce costs (Ohmi 1996, Olson *et al* 2000). Novel processes for surface cleaning are being continually developed and include the use of ozonated chemistry (de Gendt *et al* 1998, Cumpson and Seah 1996), a dry laser cleaning process (RAL 1997/98) and the use of adhesive tape to remove surface particles (Sugino and Mori 1996).

The importance of drying in the cleaning process has been discussed by Mishima and Inaba (undated) with an assessment of the relative merits of spin drying, isopropanol (IPA) vapour drying and blow drying using nitrogen gas on silicon wafers. In the

¹ Many references in this report are company brochures, leaflets, *etc.* and do not have a date of publication.

Marangoni drying process surface tension gradients in a thin aqueous film are used to induce a film of water to flow off the surface and leave it completely dry (Britten 1997, Steag Industrie AG 1993). A simple acceptable method for evaluating the cleanliness of glass surfaces has been proposed by Anghel and Chetwynd (2000) which involves measuring the geometry of a droplet of pure water dropped onto the cleaned surface.

3. CLEANING PROCEDURE AT NPL

A preliminary investigation of the sample using a microscope with a Nomarski lens attachment is used to determine if the sample needs to be cleaned. If the sample is free from surface dirt and grease or other visible contaminants surface texture measurements may be made without cleaning. The cleaning procedure adopted at NPL involves the use of ethanol, acetone and aqueous detergents and conforms to health, safety and environmental standards governed by existing legislation (Health and Safety at Work Act 1974, COSHH Regulations 1998, Environmental Protection Act 1990). The detergent used for cleaning glass is Decon 75. This is highly alkaline and does dissolve glass very slowly (around 0.1 nm per hour). In the case of metal or metal coated surfaces Kodak Photo-flo™ 600 (Eastman Kodak Company) is used. All water is filtered and de-ionised with an electrical conductivity of at least 18 MΩ cm. The cleaning procedure is carried out within a chamber with a circulating air flow in a room with air filtration and temperature control of $20\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$. As a safety precaution eye protection and gloves should be worn during the cleaning procedure and any spills should be removed from the skin with running water.

The cleaning sequence for samples, taken from Leach (2000), is given below.

3.1 THE CLEANING PROCEDURE FOR GLASS

- 1) Remove all visible contamination by an appropriate means, for example, grease is removed with ethanol or acetone solvent. The working surface is never physically scrubbed - the scrubbing pad may contain abrasive particles. Any visible dirt is blown free with a filtered air line.
- 2) The pre-cleaned sample is completely immersed in a 5% Decon and water mix for 20 minutes. Non-working surfaces are scrubbed with cotton wool soaked in Decon and water mix, keeping the surfaces wet at all times. This is washed off with water. The sample is placed in a second Decon and water bath and the working surface is zero-pressure wiped with freshly broken cotton wool. Zero pressure wiping consists of pulling out a lump of cotton wool from the bundle, breaking it open and using the exposed surface of the cotton wool to gently wipe the working surface of the sample, keeping the cotton wool and sample very wet at all times.
- 3) The next part of the procedure is to remove the Decon. Decon is a powerful cleaner and a powerful surface active agent, and becomes well-attached to the sample surface. Detaching it takes time with lots of changes of water. A total water washing time of 20 to 30 minutes is sufficient.

- 4) After the water stage, the sample is dried by blowing with filtered air. The purer the water, the more successful this will be.
- 5) The dried surface will be electrostatically charged and will attract and retain dust very efficiently. Therefore, the sample is transferred to a statically charged box, for example one made from polypropylene.

3.2 CROSS CONTAMINATION

The cleaning sequence can readily be compromised by cross-contamination, for example, a dirty cleaning bath, dirty or dusty gloves, dust from clothes, *etc.*, falling on or transferring to the sample.

Dirt, in this context, includes any material outside the specification or the dissolution spectrum of the cleaning system. Cleaning systems and handling ware (including gloves) should be dedicated to the process and not used for other applications.

3.3 THE CLEANING PROCEDURE FOR METALS

The glass cleaning sequence is also used for metals but the subsequent water washing times are much shorter since Photo-flo does not adhere so well to metals.

4. EXPERIMENTAL WORK

Four nickel shims and four samples of optical glass were used for the experimental work. The artefacts were cleaned using the procedure given in section 3.1 and examined under a microscope with a Nomarski lens attachment to check the cleanliness of their surfaces.

After cleaning one glass and one nickel sample were left exposed to the air in a busy work environment for fourteen days. A further sample of each material was handled leaving finger grease across the surfaces of the artefacts. A third set of samples was dropped on the laboratory floor several times ensuring that their measuring surfaces landed face down on the floor. The remaining two artefacts were placed in individual polypropylene boxes and used as control specimens.

All samples were examined again under the microscope before repeating the cleaning procedure. The surfaces of the artefacts were initially blown with filtered and compressed air to remove any loose dirt on the surface, before being cleaned with a suitable solvent (ethanol or acetone) and finally cleaned with the appropriate detergent. The artefacts were examined under the microscope after each stage in the final cleaning procedure to ascertain how much contamination was removed by each of the processes.

5. RESULTS

The results of the experiments are given in the following table where O indicates a dirty surface, I indicates the presence of some surface contaminants and I I indicates

the surfaces were free from contaminants.

Sample identification		Cleaning Process		
		Compressed air	Solvent	Detergent
Control	Glass	I I	I I	I I
	Nickel	I I	I I	I I
Exposed to air	Glass	I	I I	I I
	Nickel	I	I I	I I
Handled	Glass	O	I	I I
	Nickel	O	I	I I
Dropped on floor	Glass	O	I	I I
	Nickel	O	I	I I

Table 1 Results of cleaning processes on the experimental samples.

Video prints of the samples were also taken at each stage of the experimental work and a selection of these are reproduced below to show the degree of contamination of the surfaces. Surfaces were judged to be fully clean (recorded as I I in table 1) if they resembled those of the control samples (figure 1).

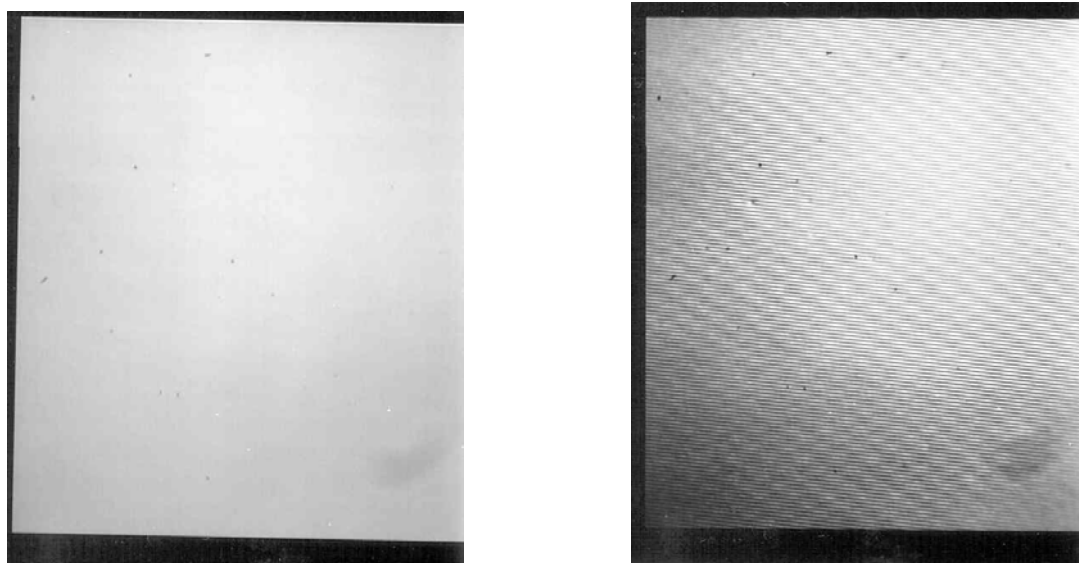


Figure 1 The glass (left) and nickel (right) control samples.

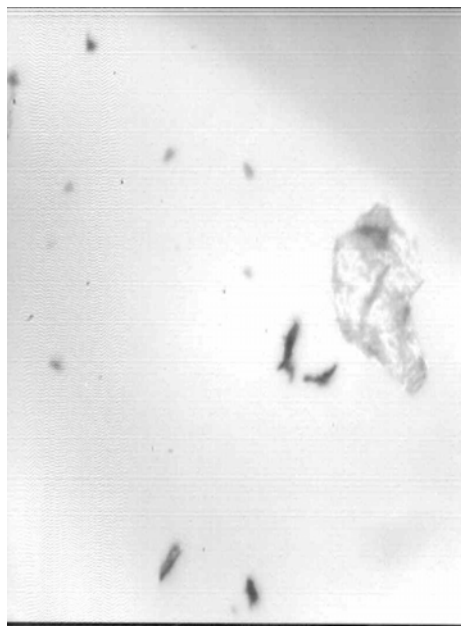


Figure 2 A glass sample showing surface contaminants after exposure to air.

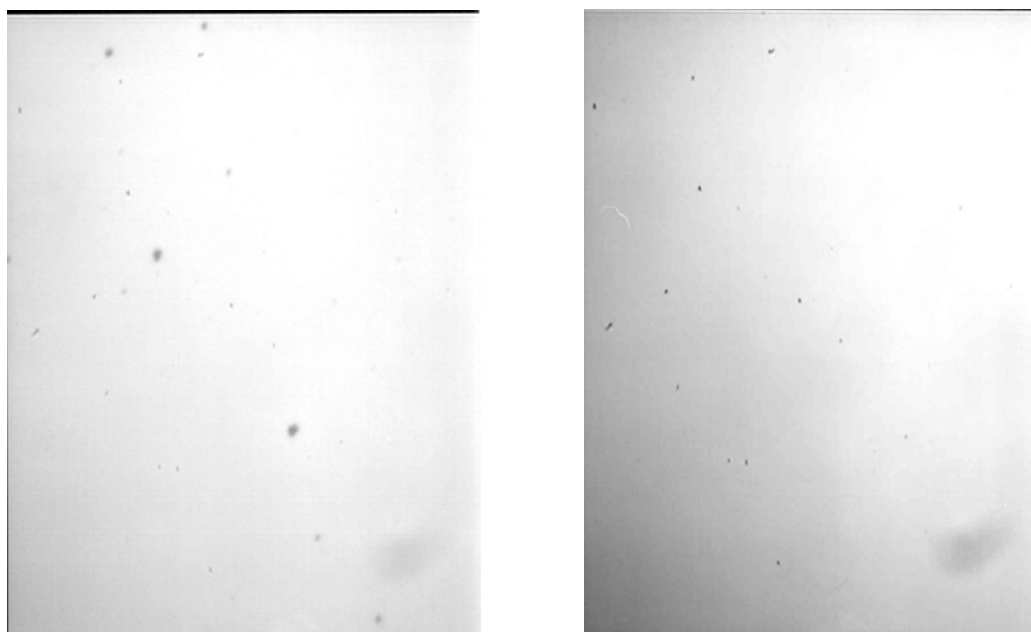


Figure 3 The same sample as in figure 2 after blowing with compressed air (left) and after cleaning with ethanol (right). The small dark spots on the right image are dirt on the microscope optics.

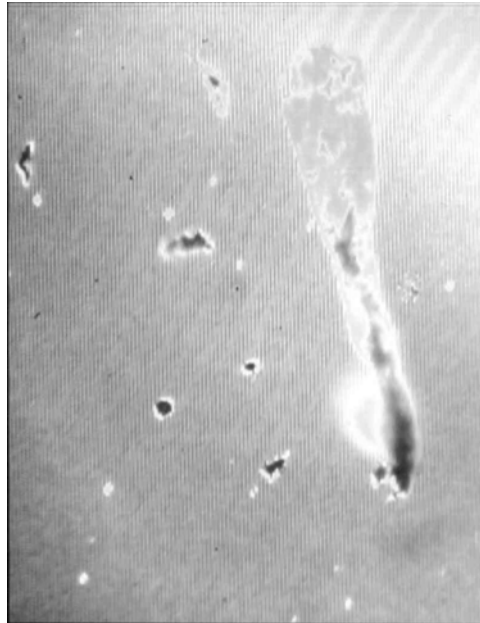


Figure 4 A nickel sample showing surface contaminants after exposure to air.



Figure 5 The same sample as in figure 4 after blowing with compressed air (left) and after cleaning with ethanol (right).

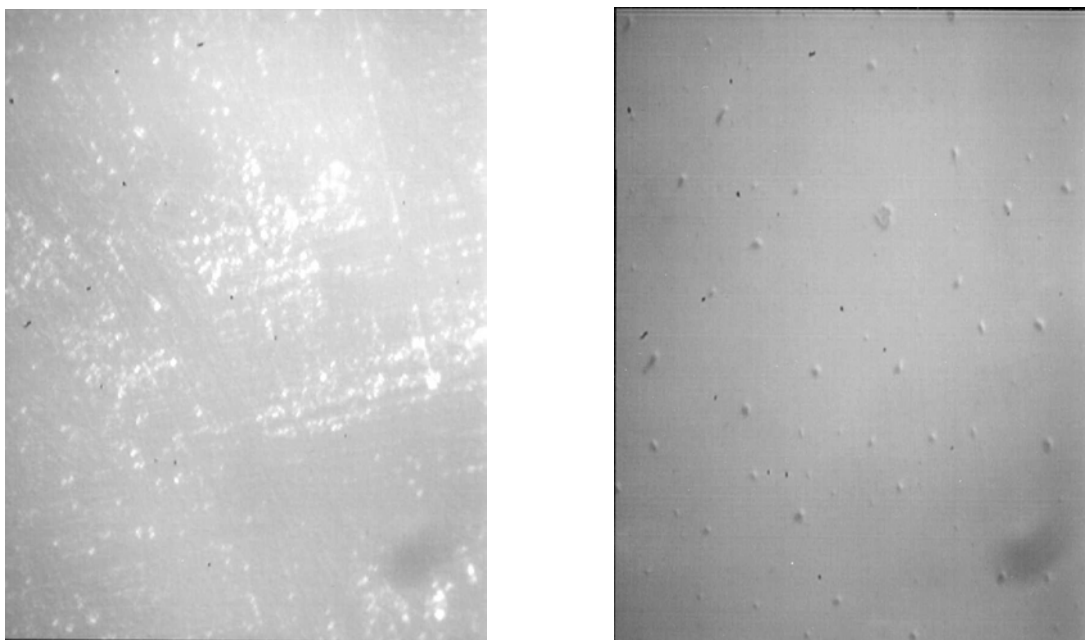


Figure 6 A glass sample showing contamination with finger grease (left) and after cleaning with ethanol (right).



Figure 7 A nickel sample showing contamination with finger grease.

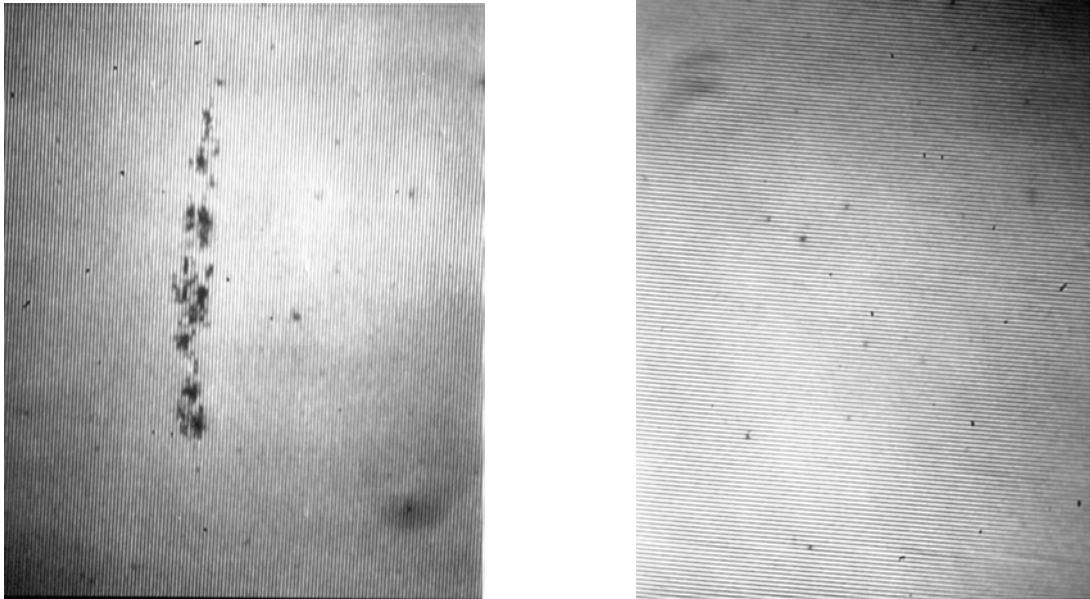


Figure 8 The same nickel sample after cleaning with ethanol (left) and detergent (right).

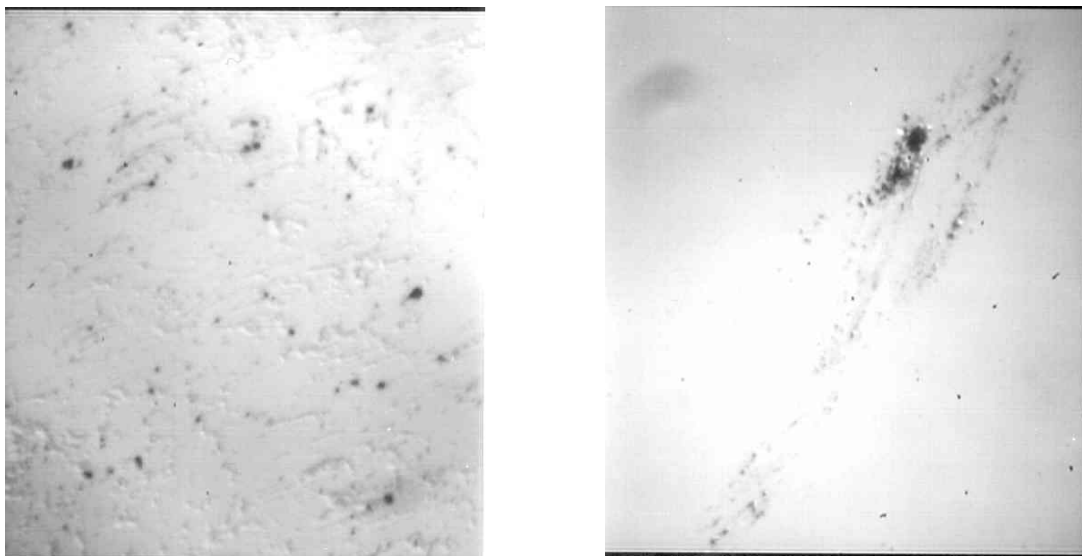


Figure 9 A glass sample showing the surface contaminated with dirt from the floor (left) and after cleaning with ethanol and acetone (right).

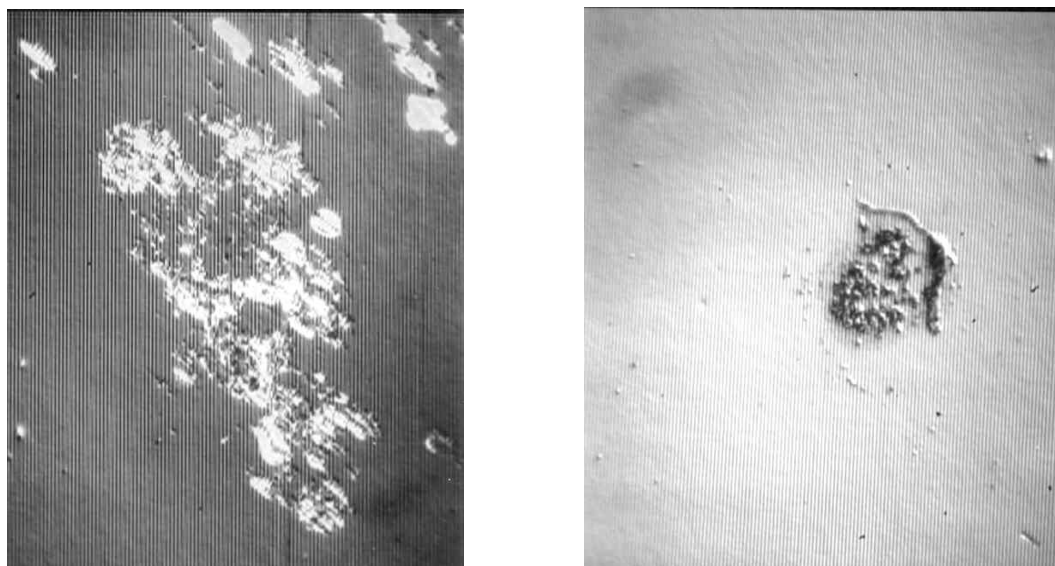


Figure 10 A nickel sample showing the surface contaminated with dirt from the floor (left) and after cleaning with ethanol and acetone (right).

6. DISCUSSION

The results from the literature search indicate that there are a number of cleaning procedures available to remove surface contaminants from a variety of materials. Many methods have been specifically designed to accommodate a large volume throughput and form part of a manufacturing process making them difficult to scale down for laboratory use. At NPL measurements of surface texture are made on small glass and nickel standard artefacts on a relatively small scale. Consequently many of the cleaning methods cited are not suitable on the grounds of health and safety concerns, cost and environmental issues.

The results of the experimental work show that, where clean surfaces have been left exposed to the air, mechanical removal of the contaminants with compressed air is not sufficient. Large particles of dirt are removed but many small particles still remain adhered to the surface. In all cases the minimum cleaning required is the combined use of compressed air and flooding the surface with ethanol.

Examination of the samples which were handled or dropped on the floor showed their surfaces to be excessively dirty, far more than would be reasonably expected under normal day to day working conditions. All these samples were successfully cleaned using the existing NPL cleaning procedures. Even on very dirty samples adequate surface cleanliness was achieved without the use of an ultrasonic cleaner. Ultrasonics is used in a number of cleaning procedures, however Anghel and Chetwynd (2000b) noted that when operated at certain frequencies damage occurred to nickel surfaces.

It is essential to have a routine inspection method for surfaces to ensure cleaning has been carried out effectively. Experience has indicated that an examination of surfaces using a microscope with a Nomarski lens attachment provides a quick and reliable method for assessing surface cleanliness. Inspection at each stage in the cleaning

process may eliminate the need for every stage to be carried out saving time and any possible damage to surfaces by further unnecessary cleaning.

7. CONCLUSIONS

1. The cleaning processes currently used in the Dimensional and Optical Team at NPL prior to measuring surface texture are suitable for their purpose.
2. Not all the stages in the cleaning process need to be carried out every time. Inspection at each stage will ensure that cleaning is tailored to the initial cleanliness of the surface.

8. ACKNOWLEDGEMENTS

The authors would like to thank Dr Val Anghel and Professor Derek Chetwynd of the University of Warwick for supplying a great deal of the references. This work was funded by the National Measurement System Policy Unit Programme for Length 1999-2002 Project 5.3.

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