

3.10 Superglue (cyanoacrylate fuming)

1. History

- 1.1 Superglue was developed in the 1950s by researchers trying to produce an acrylic polymer for the aircraft industry. It found commercial use as an adhesive system for non-porous surfaces, a main advantage being the very short cure time. Exposure to superglue vapour was reported as a possible method for the development of latent fingerprints in the late 1970s. It was reported apparently independently in Japan, North America [1] and in a private communication with the UK Home Office from Laurie Wood of Northamptonshire Police [2, 3]. Early research in the UK was carried out by the Home Office Central Research Establishment (HO CRE) investigating the relative effectiveness of a range of different commercial superglues [4] and the development of fingerprints on a range of surfaces including polyethylene, PVC and adhesive tape [5]. The use of powdering to enhance developed ridge detail was also investigated [5] and Bristol Black powder found to be most effective for this purpose.
- 1.2 The first literature publications detailing the use of the process for fingerprint development began to appear in the early 1980s [6]. Initially little was known about the reaction mechanism or the optimum treatment conditions, and fingerprint development was often slow and inconsistent, sometimes taking 24 hours to produce a developed mark. Various police forces around the world used (and some still use) the technique in a relatively uncontrolled way by treating exhibits in containers, such as fish tanks, with various proprietary cyanoacrylate adhesives. Some experimented with heating the glue to speed the process [7, 8]. Others proposed the use of other accelerating agents, including sodium hydroxide [9] and sodium carbonate [10] and comparative trials were reported between these techniques and a commercial system 'Hard Evidence', where vapours were released from a cyanoacrylate impregnated gel exposed to the atmosphere [11]. Commercial superglue fuming chambers began to be manufactured, with systems such as the 'Visuprint' [12] being available in 1983.
- 1.3 After finding the technique variable and somewhat unreliable HO CRE handed the work over to the Police Scientific Development Branch (PSDB) in 1982 for further investigation. It was quickly determined that humidity was playing a crucial role in the speed and sensitivity of the reaction [13]. The humidity was optimised primarily for polythene and other plastics, with a relative humidity (RH) level of around 80% being recommended [14]. This is the point at which solid sodium chloride will take up water from the atmosphere. Lower humidity levels resulted in slower and less effective development and higher humidity levels resulted in high background development. These experiments were repeated by HOSDB in 2009, with the results confirming the recommendations of the original study. Several prototype treatment cabinets with controlled humidity were built. A novel electronic humidity

control system was developed by a contractor (Nick Hartley) and by 1986 the commercial 'Sandridge' superglue cabinets were being installed in police forces [15]. This cabinet was designed to carry out development under the optimum conditions of humidity, evaporating ethyl cyanoacrylate at 120°C and venting to the atmosphere.



The prototype 'Sandridge' controlled-humidity superglue cabinet developed by the Police Scientific Development Branch.

- 1.4 The Sandridge cabinet had a capacity of approximately 0.5 m² and controlled the humidity by injection of vapour from an ultrasonic humidifier. The humidity was monitored, during the typical 15–30 minute treatment time, by wet and dry thermocouples linked to an electronic control system. It was manufactured by the Mason Vactron Company in Acton, London and was installed in most police and forensic service provider fingerprint laboratories in the UK and many across Europe, several of these still being in use today. This cabinet produced much more rapid and consistent results than the ad hoc arrangements most had been using and provided police forces with an effective and reliable

process for the development of fingerprints on many non-porous surfaces. In the late 1990s HOSDB was involved in, and funded in collaboration with the USA, the initial development of a larger cabinet with capacity of approximately 2 m² [16]. The prototype was purchased by Thames Valley Police and is still in operational use to the present day (up to 2011). The design was substantially modified after the original Mason Vactron Company was purchased by Foster and Freeman and is now marketed as the MVC5000. Cabinets of smaller capacities, the MVC3000 and MVC1000, have subsequently been developed and marketed.

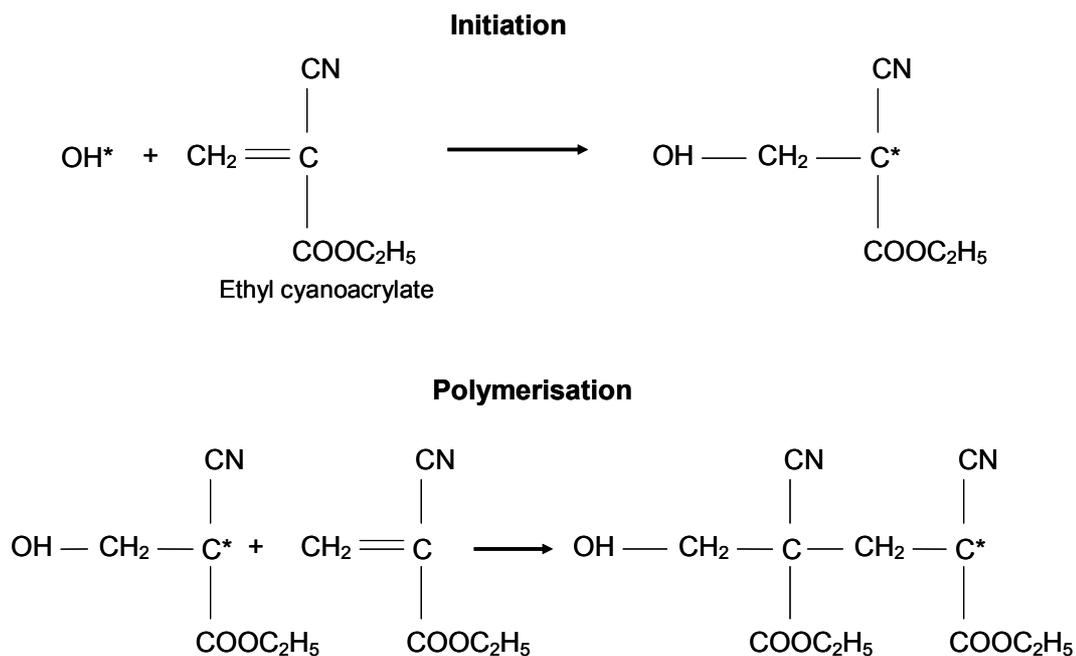
- 1.5 Superglue development is rarely used in isolation. The white deposit of a superglue-developed fingerprint can be difficult to see and photograph, especially on light coloured surfaces, although ultraviolet (UV) imaging and several of the processes described for visual examination can enhance ridge contrast. Attempts were made to improve contrast by powdering the developed ridges with coloured powders [5], but the best results were obtained by using fluorescent dyes to dye the deposit. The potential of using fluorescent dyes in this way was recognised soon after the first published papers on superglue fuming appeared. Menzel *et al.* [17] suggested the use of Rhodamine 6G (basic red 1), either by evaporation or solution staining, in combination with an argon ion laser light source. The evaporation of Rhodamine was also reported by Vaughn [18]. Stoilovic investigated Coumarin 540 as an alternative solution staining dye [19], for use with the filtered xenon lamp-based forensic light sources then under development in Australia. The high cost and limited availability of lasers and alternative forensic light sources in the early 1980s prompted investigations into dyes excited by long-wave UV [20, 21], for which cheap radiation sources were readily available. As a consequence of these studies, Ardrex was proposed for use [22] as a UV-excited alternative to Rhodamine 6G.
- 1.6 From the early 1980s through to the current time (2011), Rhodamine 6G has been one of the most widely-used dyes for developed superglue marks. Many formulations used methanol as the solvent. Methanol is extremely hazardous by skin absorption and Rhodamine was a suspect carcinogen. Alternative dye systems to Rhodamine 6G were being investigated by the mid-1980s [23]. In the mid-1980s PSDB set out to find a safer alternative which could be excited in the blue region of the spectrum and that would preferably emit in the green-yellow region. In 1985 the dye basic yellow 40 (BY40) dissolved in ethanol was identified by Sears and this was included in the manual issued in 1986 [14]. BY40 has subsequently proved to be one of the most effective dyes for dyeing marks developed with superglue, combining high fluorescent yield with low toxicity. The absorption of the dye in the violet-blue region of the spectrum and corresponding emission in the green-yellow region are particularly convenient for visualisation of developed marks. The BY40 dye has been shown to enhance the superglue-developed fingerprints to the extent that twice as many identifiable fingerprints are found on some surfaces after dyeing compared with those seen after superglue

treatment alone. The subsequent dye process is therefore an important step in sequential treatment procedures. Attempts have also been made to combine the superglue fuming and subsequent dyeing of the deposit into a single stage by co-volatilising thermal dyes that sublime [24], and investigations have also been conducted into tagging cyanoacrylates with fluorescent species. However, all these approaches have so far (up to 2011) been unsuccessful in producing as highly fluorescent marks as solution dipping.

- 1.7 A less effective water-based version of BY40 was introduced at a later date, this formulation being intended for use on surfaces where ethanol had detrimental effects (such as varnishes and some surfaces printed with inks), or in areas with poor ventilation. HOSDB subsequently reviewed the water-based BY40 formulation and investigated a further range of alternative water-based dyes [25]. The outcome of this study was the issue of a more effective water-based dye formulation incorporating basic red 14 (BR14), published for operational use in 2004. The recent development of portable, high power green lasers offers the possibility of increasing the number of fingerprints detected after staining with BR14 because the output wavelength of the laser (532nm) is well matched to the excitation characteristics of the dye.
- 1.8 Vacuum superglue fuming has been proposed as an alternative to the atmospheric, high humidity superglue development process and equipment has been developed and manufactured for this purpose [26]. Several comparative studies have been carried out between the vacuum and high humidity techniques [27, 28, 29] which demonstrated that each process has advantages and disadvantages. The high humidity technique develops marks that can be more easily seen without subsequent fluorescent dyeing and absorbs far more dye. It is therefore considered by CAST to be more appropriate to a wider range of exhibits, hence it is the technique recommended for use in the UK. However, studies into the vacuum technique have continued and refinements to the technique and equipment have been proposed [30, 31].
- 1.9 Superglue fuming, using a range of different systems, has also been used for the development of fingerprints in cars and at scenes for many years [32-35]. None of these systems have been shown to be as effective as treatment in a controlled-humidity cabinet in a laboratory (the effect of humidity and temperature is elaborated in a later sections 2.5 – 2.9, but undoubtedly they have a role in the development of fingerprints on surfaces that cannot be powdered or recovered to a laboratory. PSDB conducted an evaluation of the SuperFume system produced by Foster and Freeman [36], which came to similar conclusions to earlier studies. The SuperFume process has been successfully deployed at scenes such as hydroponics factories growing cannabis within the UK, but the suitability of the surfaces for powdering should also be considered prior to use because in many cases this may give better results.

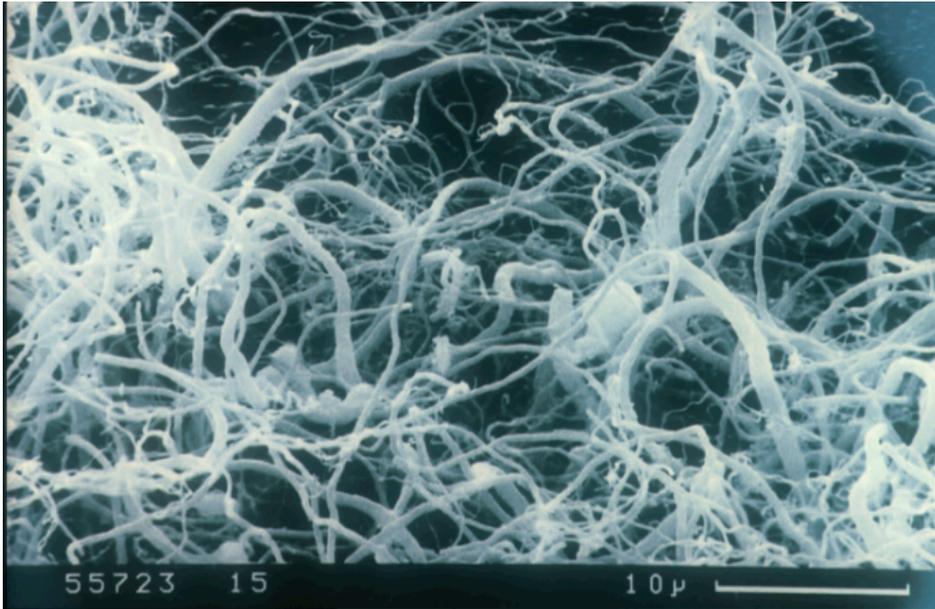
2. Theory

- 2.1 Marks developed by superglue become visible because white deposits are preferentially formed on fingerprint ridges during treatment. These white deposits are polycyanoacrylate, formed by the polymerisation of the cyanoacrylate monomer. The polymerisation reaction for ethyl cyanoacrylate is shown below; a similar mechanism occurs for methyl cyanoacrylate, which is used as an alternative by some practitioners.



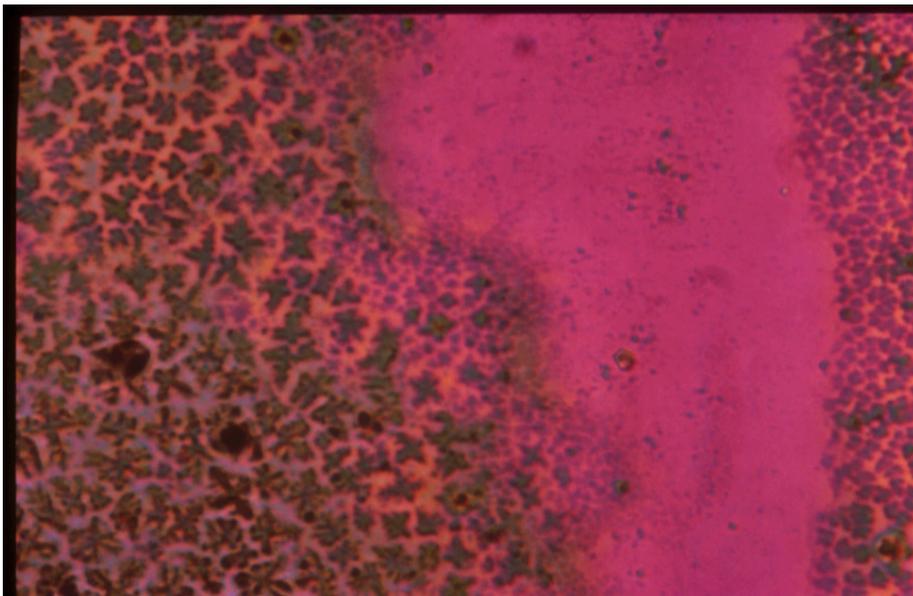
Polymerisation reaction for ethyl cyanoacrylate

- 2.2 The precise mechanism of the growth of poly-ethyl-cyanoacrylate on fingerprint residue is unclear. Electron microscopy studies by PSDB showed the growth of long fibrous deposits when the humidity was elevated (80% RH), these were not present at lower humidity levels (40% RH). These long fibrous deposits make the developed mark easier to see by eye.



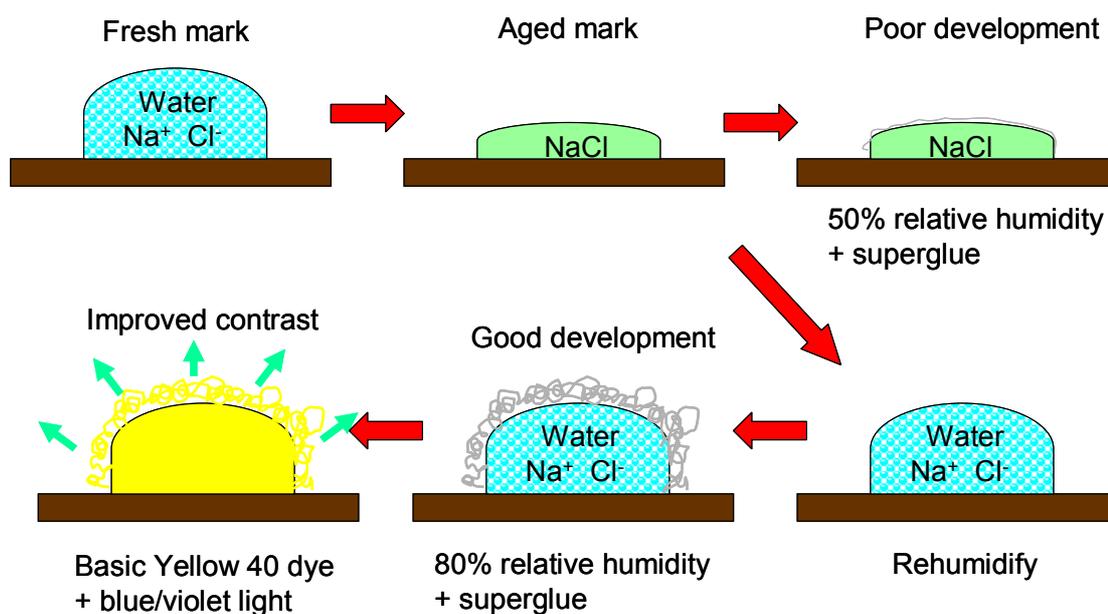
Electron micrograph of the fibrous deposits formed by superglue development at high humidity.

- 2.3 Cyanoacrylate polymerisation is base-initiated and even weak bases, such as water, will initiate polymer growth. It is believed that elevating the RH to around 80% causes sodium chloride crystals in the latent fingerprint to take up water. A saturated solution of sodium chloride (NaCl) with excess solid in a closed volume will create an RH above the solution of 75% at equilibrium. Therefore, at RH values above this NaCl crystals will absorb water from the environment around it. Similarly, any NaCl crystals in fingerprints will absorb moisture from the environment when the cabinet is set to 80% RH.



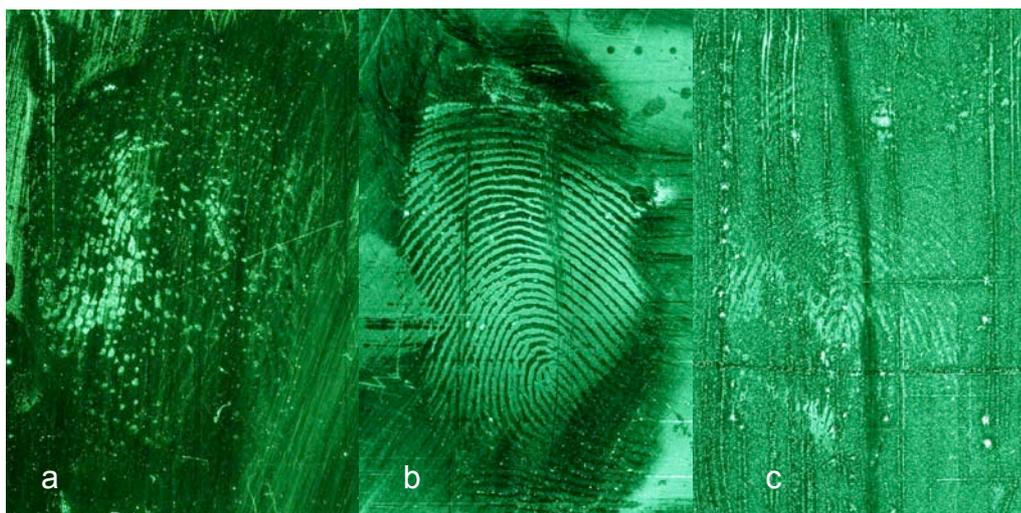
Optical interference micrograph of a dehydrated fingerprint ridge showing the formation of sodium or potassium chloride salt crystals.

2.4 This description explains one possible mechanism for polymer growth. There are undoubtedly other bases within fingerprint residues and some of these may also initiate polymerisation. Most fingerprints, however, have an initially significant water and chloride content, this is therefore likely to be a significant initiation mechanism. It is also suggested that short chains, oligomers, of cyanoacrylate may be formed due to atmospheric humidity, which may take part in further polymerisation on the fingerprint or the substrate. The superglue process is illustrated schematically below.

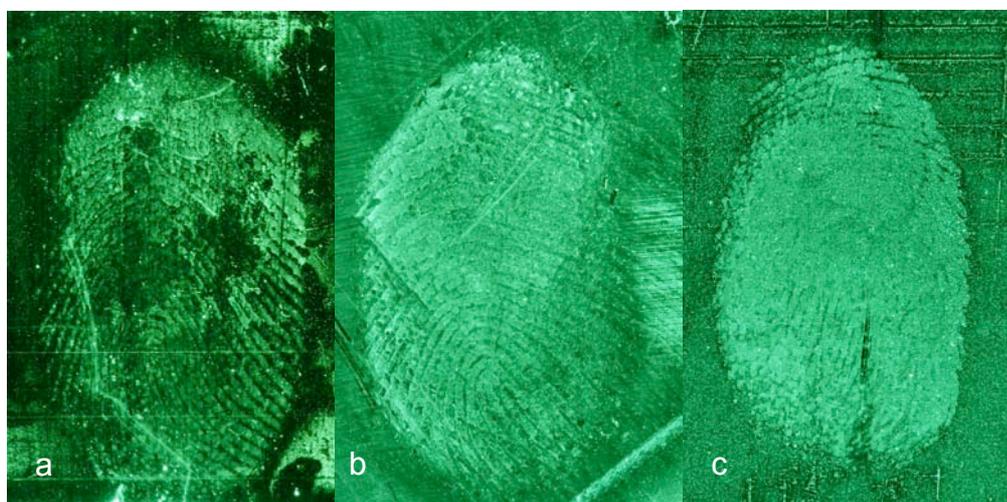


Schematic diagram of the superglue development and dyeing process.

2.5 Humidity levels below 75% RH give underdeveloped marks, humidity levels above 80% RH cause an increased background development and reduced definition of the developed mark. This can be seen in the series of photographs below, obtained for predominantly eccrine and predominantly sebaceous marks [37].



Eccrine fingerprints developed at a) 60% b) 80% c) 100% relative humidity.



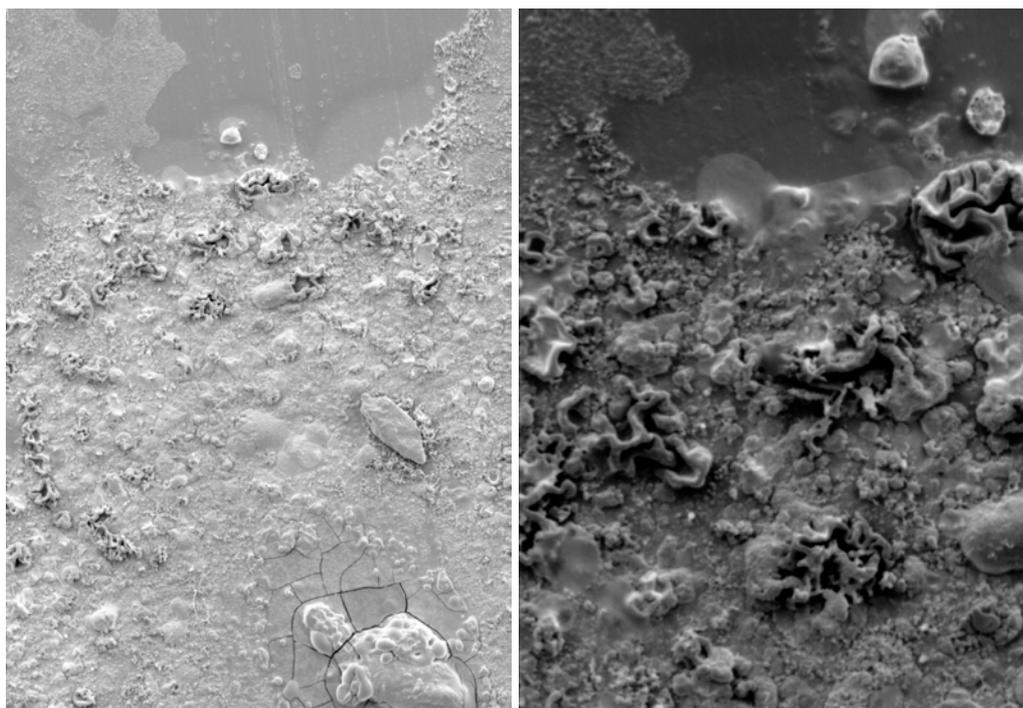
Sebaceous fingerprints developed at a) 60% b) 80% c) 100% relative humidity.

The overdevelopment at higher humidity can be better seen in the higher magnification images below.

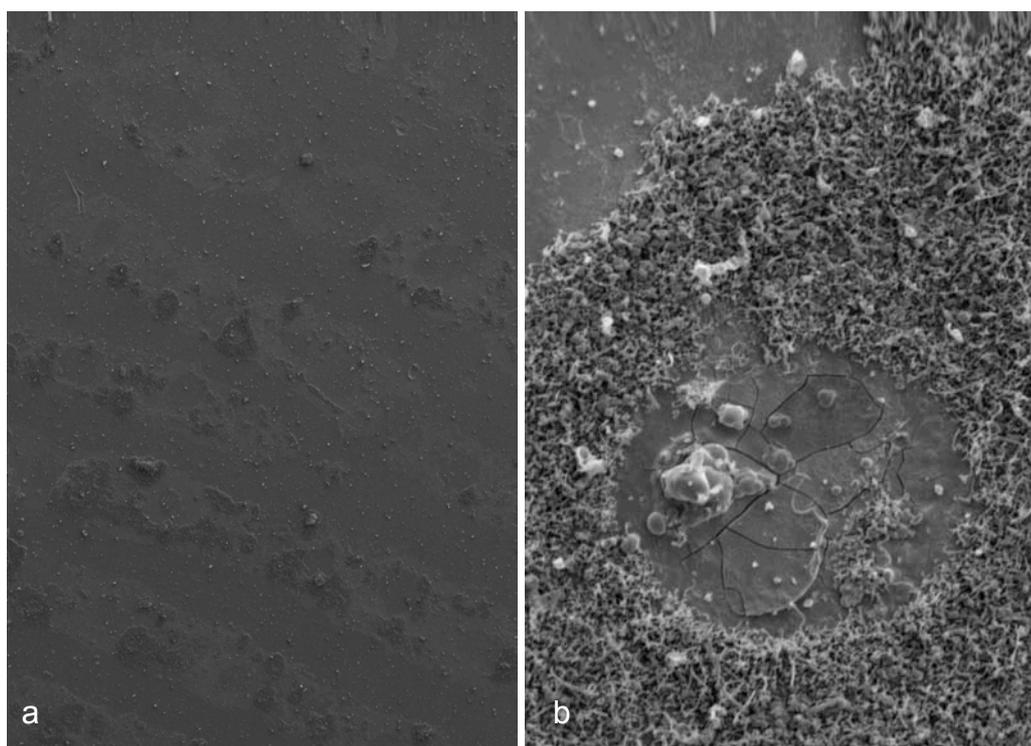


Normal (un-groomed) fingerprints developed at a) 80% and b) 100%.

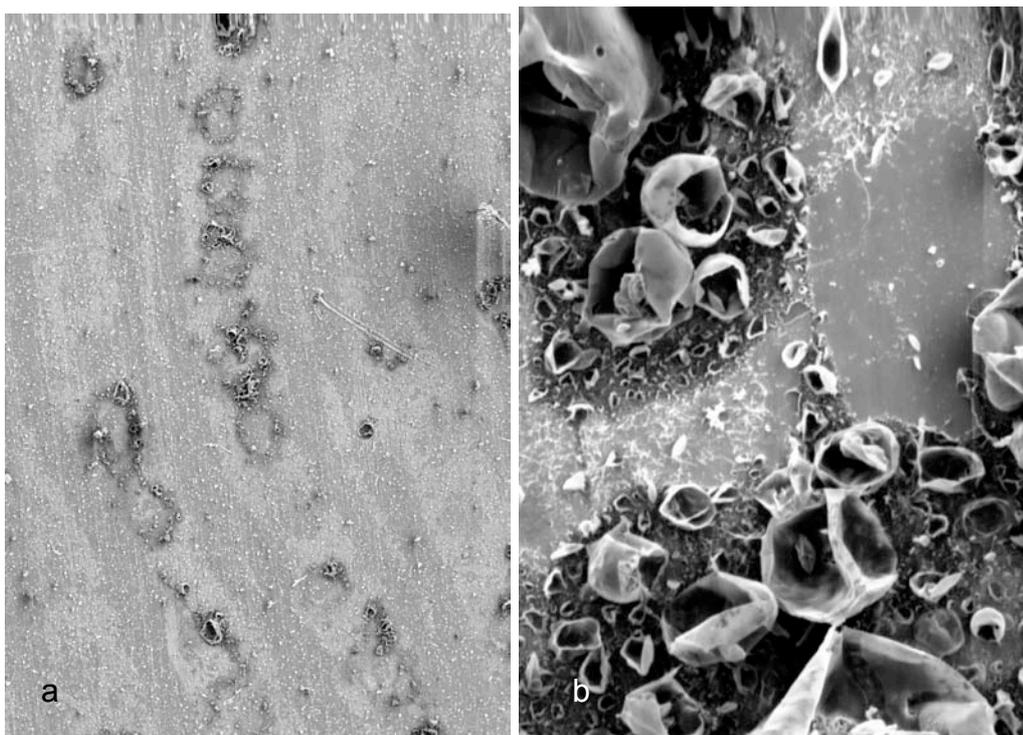
- 2.6 The different levels of superglue development for fingerprints under different conditions of humidity can be seen to be associated with different microstructures. Scanning electron microscopy has been conducted on samples developed under different humidity conditions [37], some of the results being illustrated below. It should be noted that these microscopy results are from a very limited subset of the donors used in the full study, and further investigation is required to see how consistent such observations are across a range of donors.



Fingerprint ridge developed at 60% relative humidity a) x 580 magnification b) x 1,700 magnification.



Fingerprint developed at 80% relative humidity a) x 50 magnification b) x 1,100 magnification.

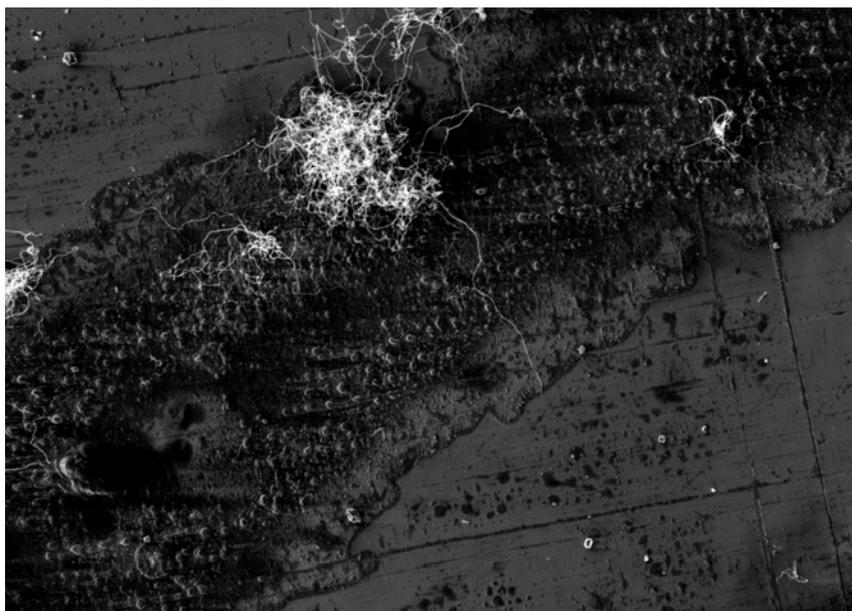


Fingerprint developed at 100% relative humidity a) x 50 magnification b) x 1,000 magnification.

- 2.7 At 60% RH, the polymer resembled a film that was gathered at some points into a tortellini-like structure. This type of polymer morphology has been observed in fingerprints developed at lower humidity by others [38]. It has been suggested that this polymer film is a result of initiation by a hard anion, leading to rapid initiation and many active centres of polymer growth and hence the polymer grows in many directions, producing a two-dimensional film [39].
- 2.8 Fingerprints developed at 80% RH had less polymer development between the ridges than those developed at 100%. There was a very high concentration of noodle-type polymer in the ridges, with a particularly high concentration around the pores. This is thought to be because the concentration of eccrine secretion is higher in these areas. It has been suggested that this type of polymer is a result of slower initiation of polymerisation, leading to fewer active centres of polymer growth, and hence growth in a single direction, producing the noodle morphology [39]. It is not clear as to why initiation might be slower at 80% RH than at 60%. If anything, it would make sense for initiation to be faster at 80% because there is a higher concentration of water molecules to initiate polymerisation. It is possible that the presence of the water molecules influences how other constituents of the fingerprint initiate polymerisation.
- 2.9 Fingerprints developed at 100% RH produced an interesting morphology that seems to be unlike any observed in the literature. The polymer resembles collapsed spheres of varying size and was mostly concentrated around the fingerprint pores. The structure of the

developed marks is predominantly flat in nature for the 60% and 100% RH samples, with some isolated raised features ('tortellini' for 60% RH, and 'collapsed spheres' for 100% RH). The noodle-like structure developed at 80% RH is most effective in retaining the fluorescent dyes used to enhance the marks.

- 2.10 There has been further investigation into which constituents of fingerprint residues may be responsible for initiation of the polymerisation reaction. Lewis *et al.* [40] found that moisture in the print prior to the fuming process was an important factor in the development of fingerprints. Eccrine marks showed a marked drop-off in quality of developed marks with time, attributed to loss of moisture from the mark. In contrast, sebaceous marks showed less age-dependence. It was thought that sebaceous constituents in the print could retain moisture in the residues, but these constituents were not, in themselves, responsible for initiating the polymerisation reaction. In the recent study conducted by CAST and London South Bank University [37] to investigate the effects of RH on fingerprint development, the microstructure of purely sebaceous marks developed at 80% RH was found to differ considerably from eccrine and 'normal' marks, suggesting a different mode of polymer growth.



Sebaceous fingerprint developed at 80% relative humidity (x 250 magnification).

- 2.11 In sebaceous marks developed using superglue, there is a large amount of spherical polymer throughout the ridge, as well as clumps of noodle-type polymer, presumably where some eccrine material is present on the ridge. The edge of the ridge shows where the oily material has spread outwards. It has been suggested that the capsule-type polymer morphology is a result of emulsion polymerisation, with fatty acids acting as emulsifiers of aqueous and oily phases [40]. The presence of small clumps of noodle-type polymer would seem to suggest that whatever is

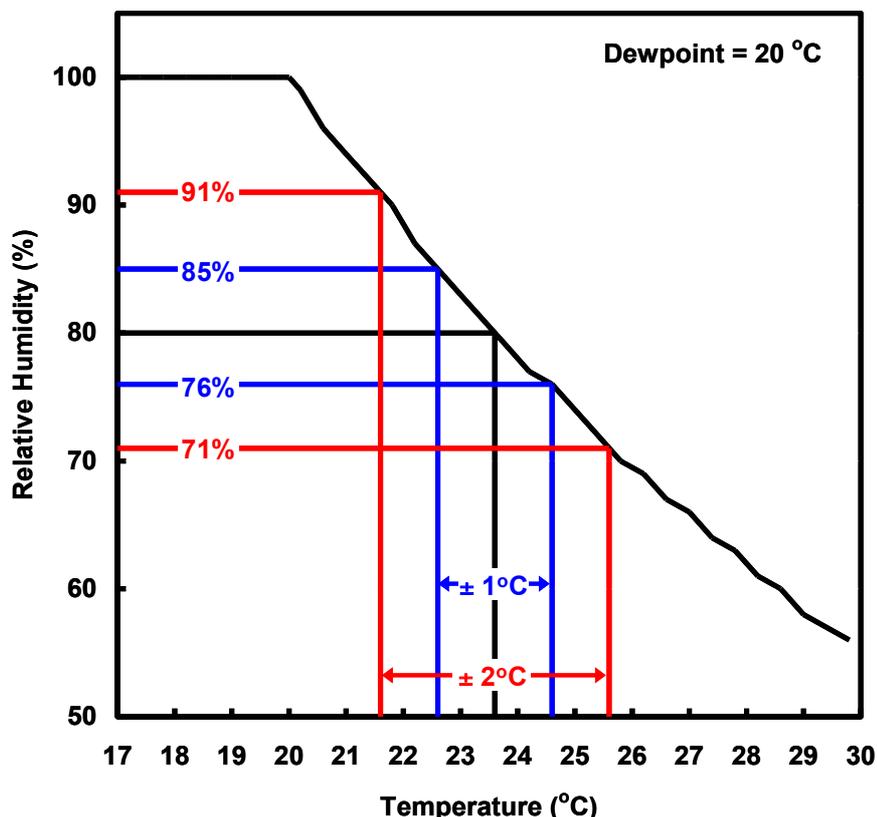
initiating noodle-type growth in eccrine fingerprints is present in unevenly distributed, smaller quantities in sebaceous fingerprints.

- 2.12 More recent research [41] looked at the role of fingerprint constituents initiating polymerisation and indicated that some of the components of eccrine sweat (lactate and alanine) were both capable of initiating the polymerisation reaction, in both cases initiation occurring via the carboxylate functional group. There was no evidence that the amine functional group in alanine played any role in polymerisation. However, the fingerprint environment is a complex one and it may be that the presence of combinations of constituents is actually more important than individual constituents in the initiation process.
- 2.13 With regard to the selection of dyes for the enhancement of developed superglue marks, it should be noted that many of the most successful dyes are basic in character. It is believed that basic dyes work better in this application because they form weak Van der Waals bonds with the polycyanoacrylate 'noodles' formed during development, predominantly due to weak binding of the dye cation with the anions associated with cyanide (CN⁻) groups in the polymers strands.

3. CAST processes

- 3.1 The CAST procedure recommends the use of a controlled-humidity superglue cabinet, four of which are known to comply with the technical specifications devised by CAST. These are the Sandridge cabinet, produced by the original Mason Vactron Company and still in use in some fingerprint laboratories, the MVC5000 and MVC3000 systems produced by Foster and Freeman (the company that purchased Mason Vactron), and the superglue cabinet produced by Labcaire. There are other systems on the market that may meet specifications, but CAST has not carried out an evaluation of them.
- 3.2 The process involves first raising the humidity in the treatment chamber to 80% RH. This value has been found by empirical testing to give a visible white deposit on the developed fingerprint and minimal background staining for the typical ambient temperature range experienced in the UK.
- 3.3 Once the humidity in the chamber has reached the required level, superglue is evaporated from an aluminium pot placed on a heater and heated to ~120°C to speed evaporation. The amount of superglue used varies between different types of chamber depending on capacity, but is selected to give a sufficient concentration of superglue vapour in the atmosphere to allow the polymerisation reaction to proceed to the extent that marks are visible. The quantity actually used should be optimised for a particular cabinet configuration by observing the quantity of residue left in the aluminium pot and adjusting it to ensure very little excess remains at the end of the cycle.

3.4 Obtaining a constant temperature within the closed treatment chamber is essential in order to maintain a constant RH because of the relationship between the two variables in a situation where air currents and fans carry air around a closed system. A theoretical plot derived from a known mass of water contained in the air at 80% RH and 20°C is illustrated below [36].



Relationship between temperature and humidity in a closed system.

- 3.5 It can be seen that small fluctuations in temperature can have appreciable effects on the local RH, and it is therefore essential to ensure that the temperature profile within the treatment chamber is as even as possible. Within the old ‘Sandridge’ style cabinet, the heat from the light bulbs used in the chamber was observed to cause fluctuations in temperature (and therefore RH), and a change to ‘low energy’ bulbs was recommended to overcome this [42].
- 3.6 The use of low viscosity (unthickened) ethyl cyanoacrylate is thought to give better results than those including thickeners. Methyl cyanoacrylate seems to give similar results to the ethyl system.
- 3.7 CAST recommends that initial photography of any visible marks be carried out after superglue treatment and prior to proceeding to treatment with a fluorescent dye stain. This is because some marks may be degraded or destroyed by the dye process and to maximise evidence recovery all marks should be recorded before dyeing.

- 3.8 The primary fluorescent dye recommended by CAST in the 2nd edition of the *Manual of Fingerprint Development techniques* [43] is BY40, dissolved in ethanol. Fingerprints dyed with BY40 are best visualised by illuminating them using the violet/blue (400–469nm) excitation band of a Quaser light source (or equivalent) and viewing the resultant blue/green fluorescence through a Schott glass GG495 filter (which has a 1% ‘cut-on’ limit at 476nm). BY40 is selected because trials by CAST in the 1980s (for which original data are no longer available) have shown it to be at least as effective in terms of fluorescence intensity as Rhodamine 6G. BY40 is preferred by CAST because it has been demonstrated not to have any of the issues of suspect carcinogenicity associated with Rhodamine derivatives. With the recent development of a blue laser operating at 460nm, it may be possible to increase the number of marks detected after dyeing with BY40, although no dedicated study has yet been carried out.
- 3.9 The dye basic red 2 (Safranin O) was also recommended for use by HOSDB. In comparative studies in the 1980s (for which original data are no longer available) it was found to be slightly less sensitive than BY40 but is excited by the green (473–548) excitation band of the Quaser light source and has an orange fluorescence that is viewed through a Schott glass OG570 (‘cut-on’ 549nm) filter. This may be a useful alternative dye for situations where the background fluoresces when illuminated by violet/blue light and obscures the developed mark, although BR14 is now preferred for this role.
- 3.10 The solvent recommended for the dyeing of superglue marks is ethanol. This is selected because it is non-toxic (unlike earlier formulations based on methanol) and has been shown to be effective in delivering the dye into the polymer deposits. However, there are cases where the flammable ethanol-based formulations cannot be used (e.g. in a laboratory that has insufficient extraction, if the dye is being applied at a scene, in cases where ethanol is causing some printed inks to run or there is excessive dye take-up by the substrate) and a water-based BY40 formulation is recommended as an alternative. However, the water-based BY40 formulation is less effective in dyeing the fingerprints and the resultant fluorescence is markedly less intense [25]. More recently CAST has reviewed a number of alternative water-based dyes and have found the most effective of these to be BR14. A formulation for this has been issued [44] for operational use, and further improvements in performance may be possible by using this dye in combination with the green (532nm) laser. There have been some recent issues with availability of the Levercet CC carrier material for the water-based formulation and some work has recently been carried out to identify alternatives [45].

4. Critical issues

- 4.1 There are a number of critical issues associated with the superglue process as recommended by CAST.
- 4.2 Superglue development should be carried out in a closed, temperature- and controlled-humidity cabinet at an RH of 80%, because these conditions give the optimum development of marks. There is evidence that some cabinets may overshoot and do not provide close control of humidity. Ideally control limits should be determined and specified. Cabinets should be kept clean and maintained regularly.
- 4.3 Marks developed using superglue should be imaged wherever possible after superglue development and prior to dyeing. There is no guarantee that marks visible after the development process will still be present after dyeing.
- 4.4 Superglue should not be used if the surface is suspected of being wetted at any point after fingerprint deposition because the fingerprint constituents that initiate polymerisation will have been dissolved.
- 4.5 The nature of the surface needs to be taken into consideration prior to dyeing the developed marks. Some surfaces may be damaged by ethanol and require dyeing with a water-based formulation, some surfaces may be strongly background fluorescent under blue/violet light and require a dye excited in a different part of the spectrum, and some surfaces may strongly absorb dye and require the developed marks to be enhanced with another means such as powders.
- 4.6 Marks may develop on different surfaces at different rates, observation of the development process is recommended and the process should be halted if over-development begins to occur, or extended if it is felt that further development of faint marks is possible.

5. Application

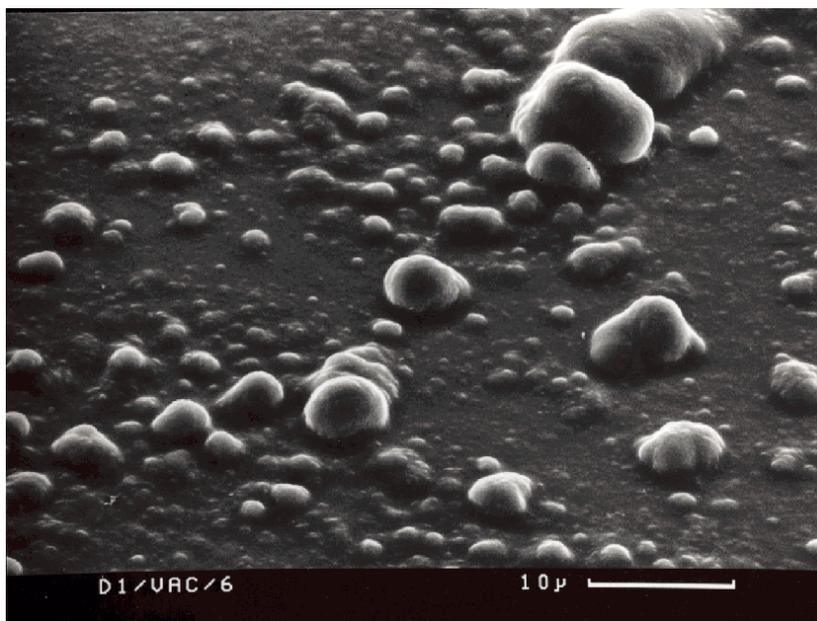
- 5.1 Suitable surfaces: Superglue is suited for use on all types of non-porous surface, including glass, plastic bottles and plastic packaging, metals, ceramics and both sides of many adhesive tapes. It is superior to powders in developing marks on surfaces that are more textured. Superglue can also be used on some 'semi-porous' surfaces, but in such situations the dyeing stage is usually omitted to prevent staining of the background.
- 5.2 The principal application of superglue is for the development of fingerprints on non-porous surfaces and adhesive tapes (although not on the adhesive side of acrylic adhesive-based tapes). It is an effective process on articles such as plastic bags, drinks cans, bottles, cowlings and vehicle number plates. Superglue generally gives better results than

powdering on textured surfaces, where powders tend to fill in the texture and clog the surface. However, the polymerisation process is thought to be initiated by water-soluble components of the fingerprint and as a consequence the process is not generally suitable for articles that have been wetted because these components are likely to have been washed away [46]. For wetted items the use of an alternative process, such as vacuum metal deposition, small particle reagent or powder suspensions, is recommended instead.

- 5.3 The technique can be effective on semi-porous items or items with glossy, non-porous coatings on porous backings (e.g. glossy magazines, printed cardboard packaging) but in these situations dyeing the article can lead to severe background staining or uptake in the porous substrate. Marks developed on these surfaces should be imaged under oblique light or UV imaging, or enhanced using a dry process such as powders or vacuum metal deposition.
- 5.4 The use of superglue fuming has been reported for fingerprints deposited on skin, but developed marks are not easy to visualise and require dye staining.
- 5.5 The method recommended for application of superglue in a laboratory is by the use of controlled-humidity cabinets. The articles to be treated are suspended or placed on shelves within the cabinet, ensuring sufficient space between them for circulation of the vapours and exposure of all surfaces of interest. Ideally, similar items should be treated together in batches. The cabinet is then humidified to the recommended level of 80% RH, and then an appropriate amount of superglue is evaporated from an aluminium foil pot on a heater at approximately 120°C. The glue cycle can be allowed to run for a set period of time, but it is best practice for the operator to watch development on the samples and halt the cycle if it looks as if overdevelopment of marks is beginning to occur. The cabinet is then placed through a purge cycle to remove fumes of cyanoacrylate vapour before the cabinet is opened and articles are removed. Articles with underdeveloped marks can be replaced into the cabinet and redeveloped. The cabinet allows several items to be treated in a single run unlike some processes, such as vacuum metal deposition, where it may only be possible to treat one item at a time.
- 5.6 If an article is to be dyed, it is immersed in a tank containing dye solution (either ethanol or water-based), then removed to a second tank containing running water until excess dye has been removed. The dyeing time for the ethanol-based dye is approximately one minute, but longer dyeing times (~ two minutes) may be required when water-based dyes are used. The article is then allowed to dry at room temperature. For larger articles, the fluorescent dye solution may be applied from a wash bottle (but never sprayed), and the dye washed off using a wash bottle, hose, or running tap water.

6. Alternative formulations and processes

6.1 The reaction of cyanoacrylates with fingerprints under low humidity and low pressure is also reported in the literature [26-31] and several comparisons have been made to the high humidity technique. In the vacuum superglue technique, the articles to be treated are placed in a chamber with a quantity of superglue, and the chamber is evacuated to a level in the region of 0.3–0.7mbar. When most of the air has been pumped out, the chamber is sealed from the vacuum pump and the superglue continues to vaporise to its room temperature vapour pressure. In general, the ‘vacuum superglue’ reaction does not give rise to the white fibrous deposit, instead it produces small beads of polymer.



Scanning electron micrograph of superglue deposit formed during vacuum superglue fuming.

6.2 The principal advantage of the technique over the high humidity process is that it is less prone to overdevelopment of marks. However, the developed marks are less easy to see and require dyeing to aid visualisation. Studies by PSDB [28] have indicated that it is more difficult to obtain dye uptake in the bead-like deposits formed by vacuum superglue and consequently it is the high humidity process that CAST has recommended.



a)



b)

Photographs showing differences between vacuum superglue development (left-hand side) and high humidity superglue development (right-hand side) a) showing over-development of high humidity mark and b) showing generally fainter appearance of vacuum developed marks

- 6.3 Comparative studies carried out by PSDB in the early 1990s [28] involved a pseudo-operational trial, dividing plastic (polyethylene) bags from high street stores into quarters. Two quarters were treated with high humidity superglue and the other two with vacuum superglue. All were then dyed with BY40 and examined using the violet/blue output of a Quaser 100. From this and parallel studies using split depletions deposited on clear polythene substrate, PSDB concluded that vacuum superglue was generally less sensitive or effective in the development of latent fingerprints. However, it should be noted that other researchers reached the opposite conclusion [27, 29] and it is recognised that both techniques have their advantages and disadvantages, vacuum superglue

being preferred where development is not closely observed and the risks of overdevelopment can be mitigated.

- 6.4 The use of superglue as a fingerprint development technique for use at crime scenes has also been investigated and several systems have been developed for the treatment of car interiors, rooms and localised treatment of small areas [32-35]. It is difficult to control accurately the humidity conditions during treatment at scenes. Consequently, where comparative assessments have been made between portable fuming equipment and treatment in controlled-humidity cabinets, the laboratory results have been superior. In 2002 PSDB assessed the SuperFume system produced by Foster and Freeman, comparing it with both powdering and superglue treatment in the controlled conditions of the MVC5000 cabinet [36]. Over 6,000 marks were deposited across a range of surfaces in a small room. Marks developed at the scene were dyed with water-based BY40, those developed using the MVC5000 were dyed with the ethanol-based formulation.
- 6.5 It was concluded that although there were a range of surfaces where superglue gave better performance than powders at a scene of crime, if the surface could be recovered to a laboratory and treated under controlled conditions the number and quality of marks developed was increased. The study concluded that scene portable fuming systems such as SuperFume do have an important role to play in treatment of scenes, in particular on textured surfaces that cannot be recovered to a laboratory. However, if articles are portable they should be taken back to a laboratory for treatment unless time and cost considerations indicate in situ treatment is preferable. The use of superglue at a scene will have health and safety implications, both in application and in the subsequent clean up. During application the fumes given off by the superglue must be contained and then safely vented, and after application vapours may still be trapped in porous items such as soft furnishings. The vapours may subsequently be released to the atmosphere, and there is a possibility that superglue deposits on hot surfaces can degrade to form hydrogen cyanide, carbon monoxide and carbon dioxide. It should also be noted that the water-based BY40 dye used in this study has since been superseded by a water-based BR14 formulation and the effectiveness of treatment at scenes should now be improved because the water-based BR14 dye gives more intense fluorescence than water-based BY40, and will be particularly effective if used with the higher power 532nm laser.
- 6.6 With regard to the dye systems used in combination with superglue, several alternatives to BY40 have been proposed in the literature. A summary of these is given to below, together with some comments about why they are not currently (2011) recommended for regular operational use by CAST.

Dye	Excitation band (nm)/colour	Viewing filter cut-on (nm)/ fluorescence colour	Comments
Rhodamine 6G (Basic red 1)	495–540 (green)	549 (orange)	Unconfirmed health and safety concerns. No better than BY40
Safranin O (Basic red 2)	473–548 (green)	549 (orange)	Recommended in CAST manual 2nd edition, but less sensitive than BY40
Ardrox	365 or 435–480 (UV or blue)	476 (blue/green)	Health and safety issue with prolonged use of UV-A
Basic red 28	470–550 (green)	549 (orange)	Not tested by CAST
Liqui-drox	365 (UV)	415 (blue)	Health and safety issue with prolonged use of UV-A
MBD	415–505 (blue/green)	515 (yellow)	Not tested by CAST
Nile Red	450–560 (green)	549 (orange)	Not tested by CAST as superglue dye
Thenoyl europium chelate (TEC)	365 (UV)	593 (red)	Health and safety issue with prolonged use of UV-A. Tested vs. BY40 by CAST, found to be inferior
MRM 10 (Rhodamine 6G, BY40, MBD)	430–530 (blue/green)	549 (529) (yellow/orange)	Not tested by CAST
RAM (Rhodamine 6G, MBD, Ardrox)	415–530 (blue/green)	529 (yellow/orange)	Not tested by CAST
RAY (Rhodamine 6G, BY40, Ardrox)	450–550 (green)	549 (orange)	Not tested by CAST

Some fluorescent dyes reported for use with superglue [19,20,21,23,47,48].

6.7 Not all the above dyes are fully soluble in ethanol, and other combinations of solvents may be recommended.

- 6.8 In order to minimise background fluorescence and improve contrast between ridges and the background, approaches to maximise the shift between excitation band and emission wavelength have been investigated. Successful approaches have included the use of dye mixtures [47], where energy transfer occurs between the excited states of the combined dyes and emission occurs at the longest wavelength from illumination at the shortest excitation band, and of thenoyl europium chelate, which naturally has a large Stokes shift and emits in the deep red/near infrared after excitation in the long-wave UV [48]. These approaches can be used operationally if it is not possible to distinguish any of the recommended dyes against background fluorescence.
- 6.9 CAST has researched alternatives to the water-based BY40 formulation, the most promising candidate systems being BR14 and Disperse Yellow 82 [25]. Disperse Yellow 82 proved difficult to dissolve into the water/carrier mix and therefore only the BR14 formulation was taken forward. The resultant formulation proved more intensely fluorescent than the water-based BY40 formulation, but not as intense as BY40 in ethanol. The effectiveness of BR14 in ethanol is closely equivalent to BY40 in ethanol and could be substituted for it, especially if used in combination with the new generation of scene-portable 5W green lasers emitting at 532nm.



Comparison of water-based basic red 14 with a) water-based basic yellow 40 (right), showing higher intensity of basic red 14 and b) ethanol-based basic yellow 40 (left), showing higher intensity of basic yellow 40

(this image should be viewed electronically to see the true intensity levels).

- 6.10 Simultaneous processes for fuming and dyeing in a single step have been investigated [24] and in some cases carry-over of the coloured or fluorescent dye into the developed ridges has been achieved. However, to date (2011) the resultant fluorescence has not been comparable with that obtained in a two-step process and the approach is not recommended. The approaches that have been considered include co-evaporation of a coloured or fluorescent dye with the superglue, and tagging of the monomer molecules with fluorescent species. This has been difficult to achieve because it is hard to get the monomer and dye to evaporate at equivalent rates, and tagging the molecules generally increases molecular weight and increases the temperatures required for evaporation.

7. Post-treatments

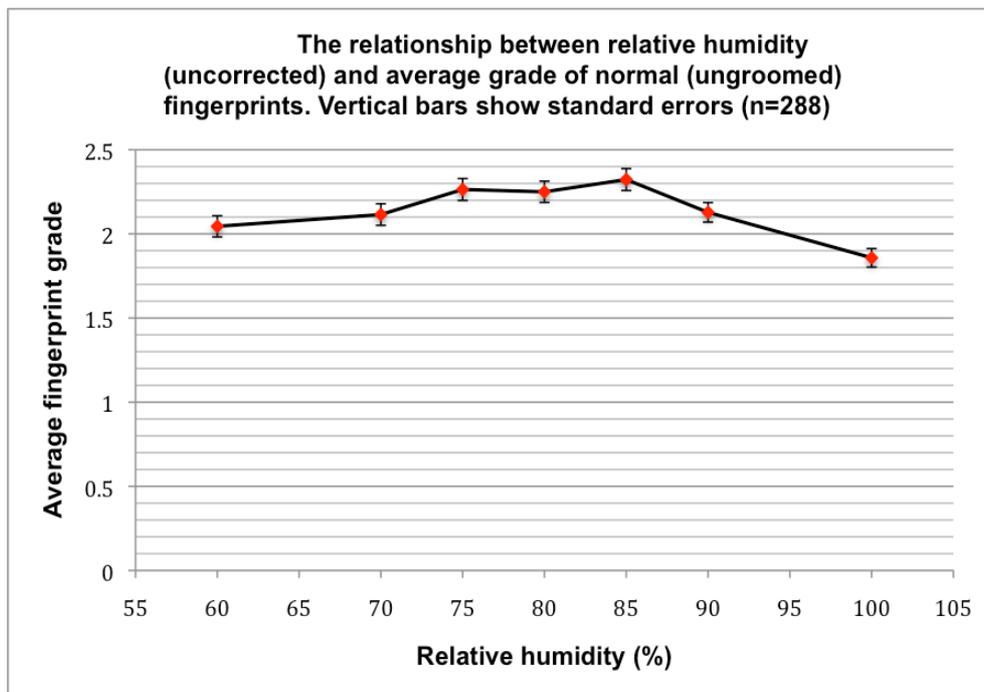
- 7.1 There are several post-treatments that can be applied to marks developed using superglue in order to improve their visualisation. The application of fluorescent (or indeed coloured) dyes has been discussed above; these are most often applied as solutions but sublimation has also been investigated. The intention is to stain selectively the fingerprint ridges to enhance their contrast with the background.
- 7.2 For marks on surfaces that cannot be solution-dyed, powdering is a possible alternative. Powders may also selectively adhere to developed areas of ridge detail although early trials indicated that not all powders are effective and some trial and error may be required to identify the most appropriate powder to use. Reasonable results have been reported with Bristol Black and black magnetic powder [5], but these are by no means the only powders to use. Powdering may also destroy marks and photography should be carried out before powdering if possible.
- 7.3 Oblique lighting and UV imaging [49] have also been used to improve the contrast between the ridges and the background. In both cases the scatter of incident light from the rough texture of the developed ridges is used to discriminate the ridges from the smooth background. The advantage of both techniques is that they are non-contact. Further detail on both these techniques is given in Chapter 4.1 Ultraviolet imaging and Chapter 2.1 Visual examination.
- 7.4 Another technique that may be used to separate superglue developed marks from patterned backgrounds is lifting using black gelatine lifters. These pick up a loose surface layer of white superglue deposit that can then be easily visualised against the black glossy background of the gel. Where marks have been dyed there is also a limited amount of dye carry-over and the lifted marks can also be viewed by fluorescence.

8. Validation and operational experience

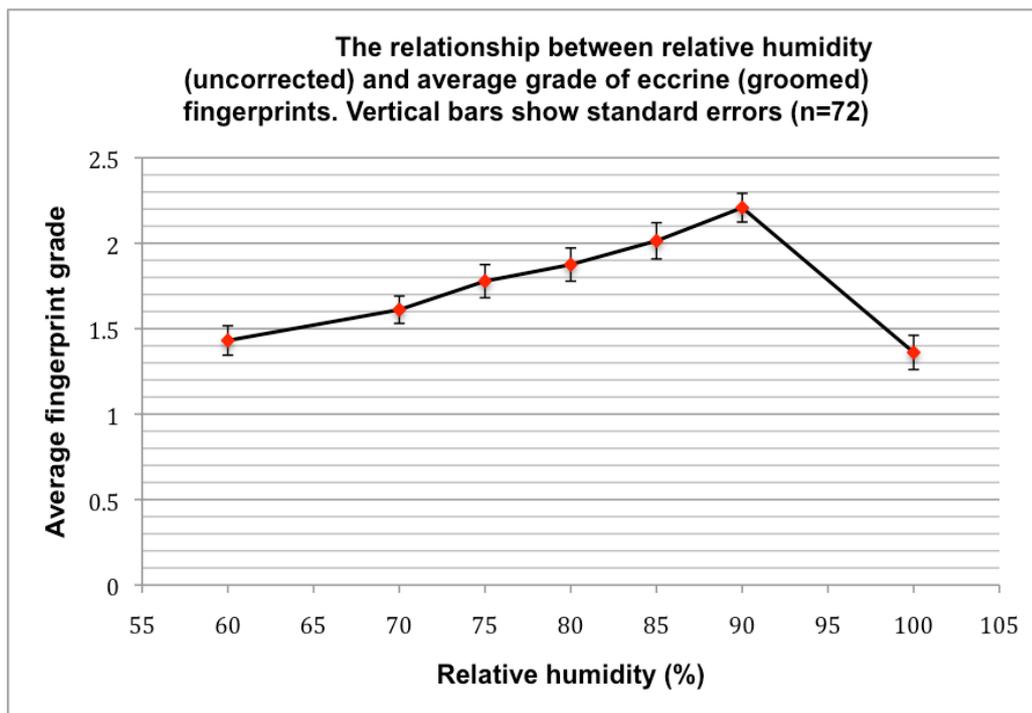
8.1 The effectiveness of superglue has been compared with that of a range of other techniques recommended for use on non-porous surfaces in a series of laboratory and pseudo-operational studies conducted by CAST from the mid-1980s to the present (2011).

8.2 Laboratory trials

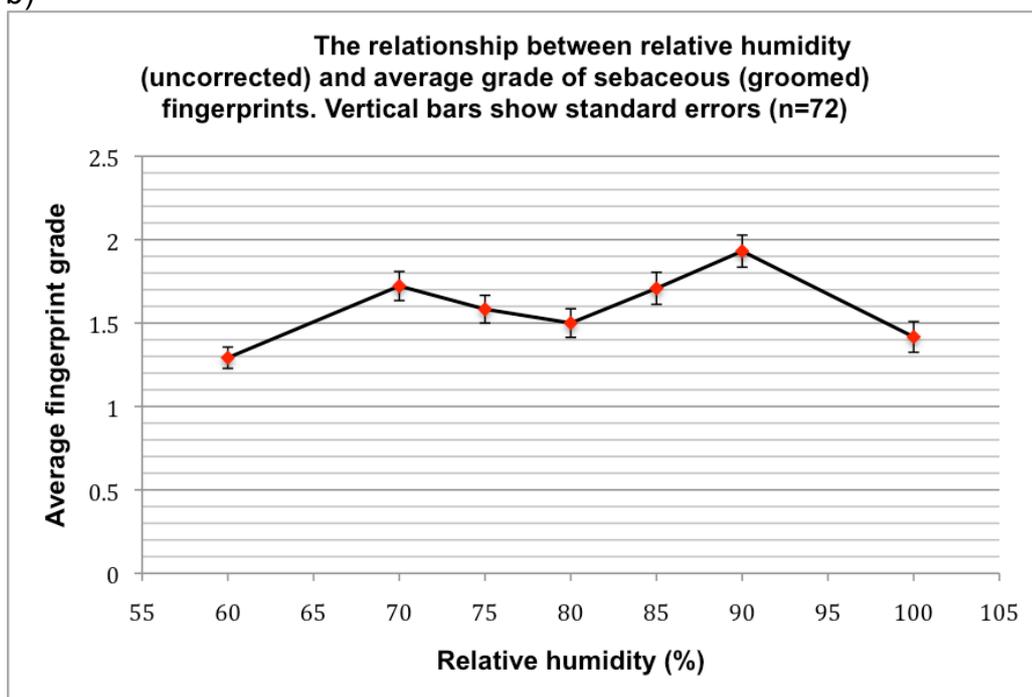
8.2.1 Laboratory trials were first conducted by HOSDB in the mid-1980s to establish the optimum conditions of RH for development of fingerprints. Unfortunately, the results of these trials no longer survive for inspection and the work was repeated in 2009 to re-validate the recommendation for 80% RH during processing [37]. The results of developing and grading 2,016 'normal' marks, 502 'eccrine' marks and 502 'sebaceous' marks are illustrated below.



a)



b)



c)

The effect of relative humidity on the quality of marks developed using superglue a) normal prints b) eccrine prints and c) sebaceous prints

8.2.2 It can be seen that the quality of the marks developed at the extremes of RH investigated, 60% and 100%, were inferior to those developed between 70% and 90% with the optimum actually being between 85% and 90%. The lower value of 80% RH is chosen for operational work because it gives some margin of error during processing and is not too close to 100%.

8.2.3 Laboratory trials have also been carried out to compare the effectiveness of superglue with both powders and powder suspensions on a range of substrates [50,51].

8.2.4 Laboratory trials were carried out in 2003–2004 to establish whether any clear recommendations could be made regarding the use of superglue or powders on non-porous surfaces [50]. A two-way trial was conducted on a range of textured surfaces comparing superglue and subsequent BY40 dyeing, and powdering with black magnetic powder. A subsequent three-way trial was performed on smooth surfaces, comparing aluminium powder, black magnetic powder and superglue, and BY40 dyeing. Both of these studies included marks of ages one day, one week and one month. Almost 10,000 marks were deposited and graded during these trials.

8.2.5 In the trial on textured non-porous surfaces 12 different surfaces were studied, including a range of laminates with different effect facings (e.g. marble, wood, granite), stone floor tiles, uPVC, computer casings and kitchen unit material. The summary of grading over 7,500 marks of all ages on all substrates is recorded in the table below.

Grade of fingerprint[Process	
	Black magnetic powder (%)	Superglue + BY40 (%)
0	12.04	16.93
1	29.61	29.19
2	19.09	16.48
3	26.26	21.08
4	13.00	16.32
Total % grade 3 + 4	39.26	37.40

Results of initial comparative experiments between superglue and powders.

8.2.6 The results obtained by the two techniques are closely equivalent, although when the results were analysed surface by surface, it could be seen that superglue developed more marks of high quality on the rougher surfaces and on older marks. It was therefore concluded that the techniques should be given equal weighting in updates to the sequential processing charts for textured non-porous surfaces in the *Manual of Fingerprint Development Techniques* [43].

8.2.7 In the second trial on smooth surfaces four materials were used, including glass, patterned and white ceramic tiles and smooth plastic-faced chipboard. Over 2,000 marks were graded in this exercise. The results are summarised in the table below.

Grade of fingerprint	Process		
	Aluminium powder (%)	Black magnetic powder (%)	Superglue + BY40 (%)
0	4.6	5.7	9.7
1	20.4	20.3	24.6
2	16.5	17.2	11.9
3	22.4	30.0	25.4
4	36.1	26.8	28.3
Total % grade 3 + 4	58.5	56.8	53.8

Results of further comparative experiments between superglue and powders.

8.2.8 Overall, there was little difference between the three processes, although the observation in previous trials that aluminium powder performed best on smooth surfaces was confirmed. Powdering would marginally be the preferred process on smooth non-porous surfaces, but superglue gave closely equivalent performance.

8.2.9 The comparison with powder suspensions carried out more recently (2007) is more fully reported in Chapter 3.7 Powder suspensions. This was an extensive study looking at over 37,500 marks over 23 different surface types. There were variations in performance across individual surfaces, but general trends could be seen. These indicate that superglue and powder suspensions are closely equivalent in performance when used to develop fingerprints on non-porous surfaces, but the sequence of powders followed by powder suspensions was found to be more effective than superglue and dyeing overall [51].

8.3 Pseudo-operational trials and operational experience

8.3.1 PSDB carried out a pseudo-operational trial on 200 plastic bags in 1986, and demonstrated that superglue combined with dyeing and subsequent fluorescence examination was a highly effective process for this type of surface, although not as effective as vacuum metal deposition. The results of this study are described in greater detail in Chapter 3.11 Vacuum metal deposition. Because not all police forces had vacuum metal deposition equipment, superglue and dyeing was considered an effective alternative.

8.3.2 A comparison between vacuum metal deposition and vacuum superglue was also carried out by Misner [52], who found that vacuum metal deposition developed 180/229 (79%) deposited marks to an identifiable standard compared with 141/229 (62%) marks developed by superglue, a similar margin to that in the PSDB study. Taroni *et al.* [53] conducted a similar study but concluded that superglue and vacuum metal deposition were of a similar sensitivity. However, a further study by Masters and

DeHaan [54] again concluded that vacuum metal deposition was more sensitive than superglue on older marks (> three years) although equivalent on fresher marks (< two months).

8.3.3 PSDB conducted a comparative pseudo-operational trial between high humidity and vacuum superglue in the early 1990s [28], dividing ten plastic (polyethylene) bags obtained from high street stores and donated after use into quarters. Two quarters were treated with high humidity superglue and the other two with vacuum superglue. All were then dyed with BY40 and examined using the violet/blue output of a Quaser 100. The number of fingerprints and scraps of ridge detail developed were recorded, the results being summarised below.

High humidity superglue		Vacuum superglue	
Fingerprints	Ridge detail areas	Fingerprints	Ridge detail scraps
32	47	16	29

Results of comparative experiments between vacuum and high humidity superglue techniques.

8.3.4 From this and parallel studies using split depletions deposited on clear polythene substrate, PSDB concluded that vacuum superglue was generally less sensitive or effective in the development of latent fingerprints. PSDB found that there were problems with low dye take-up by marks developed using vacuum superglue, resulting in less developed marks being detected. However, other researchers carrying out similar studies reached the opposite conclusion [27, 29] and it is recognised that both techniques have their advantages and disadvantages. However, high humidity superglue continued to be the primary process recommended for operational use in the UK.

8.3.5 An analysis of the effectiveness of the superglue process on operational work in Essex Police Laboratory was conducted by Taylor [55] over a period of three months in 1995. Results from processing 430 items are summarised below. At the time of the work a 16 point standard was in place for fingerprint identification in the UK, and the marks were assessed by a fingerprint expert in terms of the number of 'second-level detail' features present.

Article type	>16 points	8–15 points	Items overall
Adhesive tape	3	6	18
Cash bag	0	4	46
Cowling	3	57	145
Credit cards	0	3	22
Latex gloves	0	0	16
Number[style so far] plates	2	4	42

Screwdrivers	0	0	13
Store cards	0	0	21
Sweet wrappers	0	0	11
Bullets/pellets	0	0	8
Crisp packets	0	1	7
Polythene bags	0	0	7
Miscellaneous	5	31	74

Results of operational work using superglue.

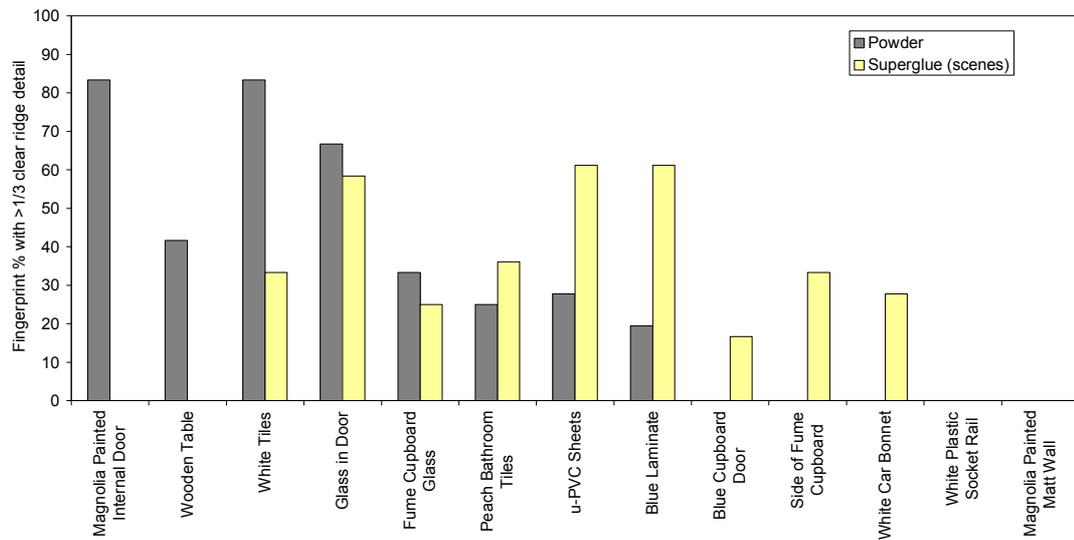
8.3.6 The miscellaneous items where the superglue process had most success included bin liners, a photograph and a telephone. The majority (91%) of items for which marks containing >16 points (the minimum fingerprint quality standard then in place) were recovered were plastics, suggesting that superglue is effective for this type of article. The effect of time before treatment on the number of marks developed was also assessed, as was the effect of any contamination present on the surface on subsequent development. Results indicated that superglue became less effective as the age of the mark increased. Development was still observed on articles contaminated by chemicals, blood or oil, but the presence of drugs residue or powder inhibited development and no marks were found on articles known to have been wetted. At this time, fluorescent dyeing was not being used as a secondary treatment; had this been the case, the number of recorded marks would have increased.

8.3.7 Subsequent operational experience has shown that superglue continues to be highly effective on plastic, non-porous items and the technique is extensively used on plastic bags and cowlings. However, some police forces had found that powders give equally good results on the inside of cowlings and it has recently been observed that powder suspensions may be more effective still. Two police forces recording data and changing from superglue to powder suspensions as a development technique on cowlings have observed increases in the number of marks developed. This is thought to be partly attributed to the fact that such items may be exposed to moist or wet environments, which are less detrimental to the powder suspension process.

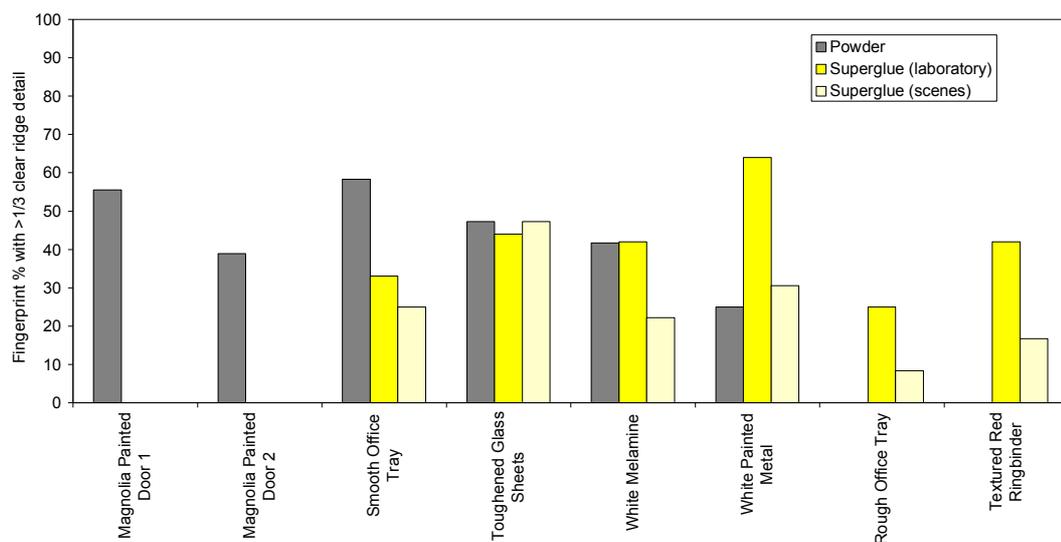
8.3.8 The operational work outlined above also shows that superglue is an effective process for developing fingerprints on adhesive tapes. Recently concluded research by CAST [56,57,58] indicates that superglue is the most effective process for developing fingerprints on the non-adhesive side of tapes, and is closely equivalent in performance to powder suspensions on the adhesive side of tapes with rubber-based adhesives. For treatment of such tapes superglue has the advantage that it develops marks on both sides of the tape simultaneously. However, on tapes with acrylic-based adhesives superglue does not develop marks on the adhesive side and carbon-based powder suspensions should be used instead. An operational trial to compare the effectiveness of superglue and carbon-based powder suspensions was carried out and

indicated that carbon-based powder suspensions were superior in this application, this being reported in Chapter 3.7 Powder suspensions.

8.3.9 Pseudo-operational trials were also conducted to establish the relative effectiveness of superglue carried out at scenes using a SuperFume unit, and those developed under laboratory conditions in an MVC5000 unit [36]. In this study over 6,000 marks were deposited across a range of surfaces in a small room. Marks developed at the scene were dyed with water-based BY40, those developed using the MVC5000 were dyed with the ethanol-based formulation. This was representative of what would be carried out at scenes and in most laboratories. The results of this study are summarised below.



Comparison of the effectiveness of superglue and powders on surfaces treated at a simulated scene [36].



Comparison of the effectiveness of superglue and powders on surfaces treated at a simulated scene, and surfaces treated with superglue in a laboratory [36].

- 8.3.10 It could be seen that SuperFume gave better results than powdering on several surfaces, although better results still could be obtained where a controlled-humidity superglue cabinet was used. The ultimate choice of technique should take into account effectiveness and time and cost considerations. It should also be taken into account that aluminium powder was used on all surfaces, whereas more recent guidelines may dictate use of an alternative powder. The results obtained from powdering in this trial are therefore less than optimum, and further work may be required to clarify this.
- 8.3.11 The most recent pseudo-operational trial that has been carried out including superglue has been the reassessment of the optimum processing sequences for plastic bags and packaging material [59]. Once again, the results are more fully reported in Chapter 3.7 Powder suspensions, but indicate that superglue followed by vacuum metal deposition may be the best processing sequence for this type of surface.

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3.11 Vacuum metal deposition

1. History

- 1.1 Vacuum metal deposition (VMD) is a long-established industrial technique for the application of metal coatings to components such as glass mirrors. In 1964, Professor S. Tolansky, working on the manufacture of interference filters at the Royal Holloway College of the University of London, noted that the deposition of silver in a vacuum system developed accidentally deposited latent fingerprints on glass optical components. An investigation into the process as a fingerprint development technique was proposed to the Home Office by Professor Tolansky. However, this was not pursued at the time by the Home Office because other techniques for fingerprint detection on glass were considered cheaper, easier to use, and sufficiently effective.
- 1.2 In 1968, it was reported by French workers [1] that VMD from a mixture of zinc, antimony and copper powder was capable of developing latent prints on paper. As a consequence of this paper, interest in the technique was revived in the UK and Tolansky initiated a research programme to investigate the optimum conditions and the potential applications for VMD. One of the early objectives of the research was to establish why the French combination of metals was effective. Closer examination of metal coatings deposited by the French laboratory indicated that the coating was almost entirely zinc, the presence of antimony and copper not being necessary to develop prints [2].
- 1.3 The research programme initiated by Tolansky [2] investigated the deposition characteristics of a range of metals on paper substrates, identifying single metals and metal combinations giving the optimum print development. Research was also carried out into the ability of the technique to detect latent prints on fabrics. These experiments showed that although some print development was obtained by the use of single metals, in general the best results were obtained by the use of a combination of metals, typically gold or silver followed by cadmium or zinc. The gold/zinc combination is currently (2011) used operationally.
- 1.4 The potential of VMD to develop fingerprints on fabrics was further explored by the Atomic Weapons Research Establishment (AWRE) under contract to the Home Office [3,4,5]. The work looked at identifying the best metal combinations for developing prints [3], transfer of both latent and developed marks onto photographic paper [3], and the effect of humidity [3,4]. The researchers considered the effect of different washing and wearing conditions on print survival [4] and expanded the study to look at synthetic fabrics [5]. It was considered that the chances of fingerprints surviving on washed and worn fabrics under 'field' conditions was small, but finite.
- 1.5 In the mid-1970s the increasing use of low density polyethylene (LDPE) carrier bags in a variety of crimes, in particular Irish Republican Army

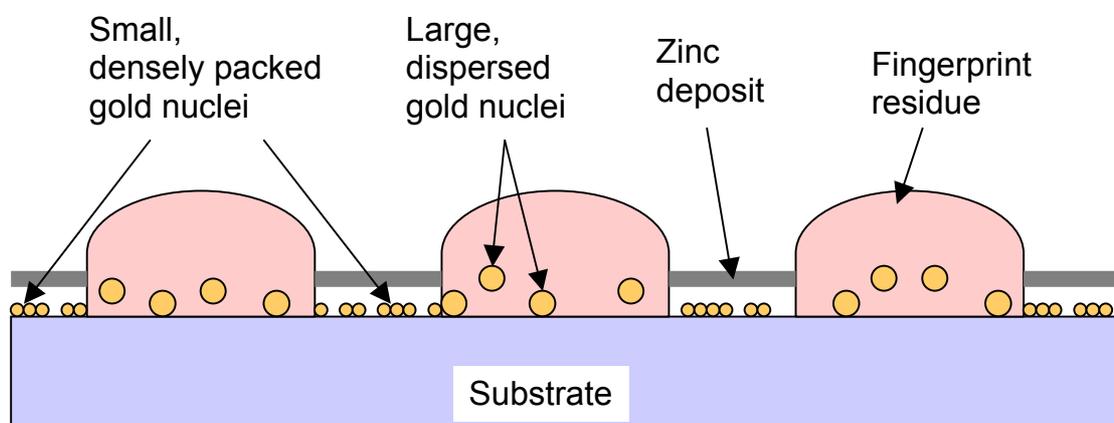
(IRA) improvised explosive devices (IEDs), led the Police Scientific Development Branch (PSDB) to look for better ways of developing fingerprints on polyethylene. A programme of work evaluating the various metal combinations on a variety of plastics and rubbers was set up and considerable success achieved using gold followed by cadmium on polythene and most plastics with the notable exception of plasticized PVC. This and all the early experimental work was carried out on small-scale equipment with 12 inch bell jar coaters. Silver-cadmium combinations gave slightly poorer results than gold-cadmium, with copper-cadmium less good. To make the system more viable for operational casework modification of horizontal 24 inch coaters was investigated and a system purchased by PSDB in 1976. This proved very successful during operational trials with police forces and over the next decade around 20 similar machines were installed [6]. Monitoring of the cadmium levels in the vicinity of the chamber and during cleaning operations in 1977-1978 indicated that figures approaching 10% of the maximum exposure level were being generated and around this time there was also a proposal to reduce the permitted levels for cadmium. Gold followed by zinc deposition was known to give similar results to gold-cadmium although deposition of zinc is slower and more difficult. The decision was made to switch to gold-zinc for all operational police systems as the maximum exposure limits for zinc were many times higher and there was no likelihood of these being exceeded in operational use.

- 1.6 From the late-1970s until the late-1990s PSDB worked with manufacturers and introduced a number of improvements including larger chambers, liquid nitrogen cold fingers and semi-automated sample loading systems. Over this time most suppliers had moved from manually operated valve systems to automated, or semi-automated, control systems [7]. Several trials were carried out by PSDB between VMD and other techniques for developing fingerprints on polyethylene including small particle reagent, superglue and fluorescence examination [8,9,10] and it was found to out-perform all of these techniques. In particular VMD was shown experimentally and operationally to develop fingerprints that had been exposed to extended water immersion, something that no other technique at the time (late-1970s and early-1980s) could cope with.

2. Theory

- 2.1 There is general agreement on the theory associated with normal development of prints by the VMD method. The reason that the metal combinations are postulated to work well is due to the condensation characteristics of zinc (and cadmium). These metals will not condense on grease, such as that found in fingerprint residues, even when these substances are only present as a monolayer. However, zinc will deposit on small nuclei of metal, and this is the reason that gold or silver deposition is carried out first. Gold and silver can be deposited over the

entire surface, and begin to form nuclei, the morphology of which depends on the nature of the surface (surface energy, chemical species present) they are being deposited on. The resultant gold coating is very thin (several nanometres only) and discontinuous. However, in the regions coated with the fatty residues of the latent fingerprint, the gold diffuses into fat and hence there are no gold nuclei close to the surface. As a consequence, when zinc is subsequently deposited, it will condense on the regions of gold nuclei (i.e. the background substrate), but not on the regions of the fatty deposit (i.e. the fingerprint ridges). This theory of nucleation was discussed in more detail by Stroud [11,12]. It should, however, be noted that there is no conclusive evidence of nuclei diffusion and it is possible that the effect observed may be solely attributable to zinc growing on regions of different nuclei size at different rates. The normal development process based on nuclei diffusion is depicted in the schematic diagram and photographs below.



Schematic diagram of normal development, showing zinc depositing where gold nuclei are available on the surface.

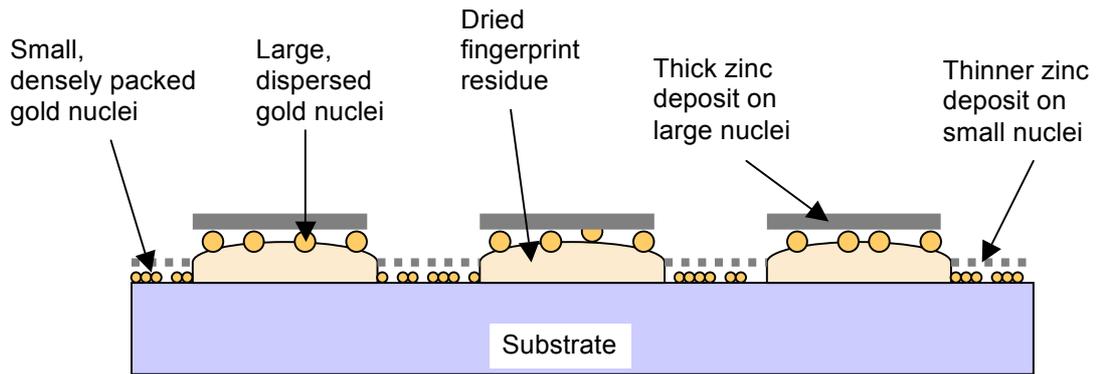


Photograph of a normally developed mark on a polyethylene bag.

- 2.2 Tests carried out to determine which components of the latent print were most likely to be responsible for inhibiting metal deposition identified several substances, including stearic acid, palmitic acid, cholesterol oleate, glycerol trioleate and amino acids L-arginine monohydrochloride, L-leucine, and DL-threonine. Most of these substances are non-water soluble or long chain fats or acids with low vapour pressure, which determines their stability and non-migration over the surface during the VMD process. These findings were in accordance with the observation that VMD was capable of developing prints on substrates exposed to wet environments, many of these substances being insoluble in water. In the late 1970s PSDB funded work at Imperial College, London using electron spectroscopy for chemical analysis (ESCA) and ion beam etching in order to establish the depth profile of gold on the surface. This was done to determine whether loss of gold nuclei into the fats of a fingerprint would account for the 'normal' development of light ridges. Experiments to study the diffusion of gold into thin films of stearic acid [13] indicated that 60% of the gold penetrated the stearic acid to a depth greater than the detection depth of the ESCA surface analysis technique, and hence would probably not be sufficiently close to the surface for zinc to nucleate on it. The work was however regarded as inconclusive as although a slight increase in gold was detected during etching down through the

surface layers it was felt that this might have been due to ‘knock on’ effects of the heavy ion etching.

- 2.3 Transmission electron microscopy has also been used to confirm that the size and distribution of gold nuclei formed during the deposition process varied greatly according to the substrate and the chemical species present [14]. Transmission electron micrographs of gold films on carbon support grids which had some deposited LDPE crystals showed few large nuclei on the LDPE compared with large numbers of small nuclei on the carbon. This confirmed that a variation in nuclei size could be produced in areas with different binding energies. Rayleigh scattering from the gold films also showed changes in colour indicating variations in nuclei size. It was this difference in nuclei size and distribution, coupled with diffusion of gold into the fatty deposits, that was believed to contribute to the subsequent delineation of the print during VMD.
- 2.4 In practice, many prints developed using VMD may be ‘reverse developed’, i.e. zinc preferentially deposits on the fingerprint ridges rather than the background. There are differences in opinion as to why this arises, the main theories being outlined below.
- 2.5 Kent *et al.* [15] attribute reverse development to absorption of mobile species of the fingerprint residue into the substrate, leaving a solid, primarily inorganic residue that acts as a preferential nucleation site for the zinc. More gold diffuses into the polymer substrate than into the solid residue, hence zinc deposits on the ridges first. Smith, as quoted by Jones *et al.* [16-18]) proposed that zinc deposits on the ridges because it is able to align crystallographically with some of the crystalline constituents in the deposit (e.g. sodium chloride) and undergo epitaxial growth. Most recently, Jones *et al.* [16-18] have proposed an alternative theory related to the types of gold nuclei formed. The gold nuclei in the ridges form at a different rate to those growing on the substrate and in the furrows, hence a regime exists where the gold film on the background has reached a state where zinc cannot nucleate, but on the ridges the nuclei are a suitable size for zinc to deposit. This theory closely relates the type of print developed to the amount of gold deposited initially.
- 2.6 Current (2011) CAST thinking is that the reverse development is due to the fingerprint deposits becoming dried out, either by air drying or by the preferential absorption mechanism outlined by Kent, or contaminated, thus inhibiting any diffusion of the gold nuclei into the fingerprint residue. The dried ridge is likely to have a higher surface energy than the background and therefore larger gold nuclei will form in these regions. These larger gold nuclei will sit on the surface of the ridge because their diffusion is inhibited and because the gold nuclei in the region of the ridges are larger, zinc deposition occurs at a faster rate. This is illustrated schematically below.



Schematic diagram of reverse development, showing different rates of zinc deposition according to size of gold nuclei available on the surface.

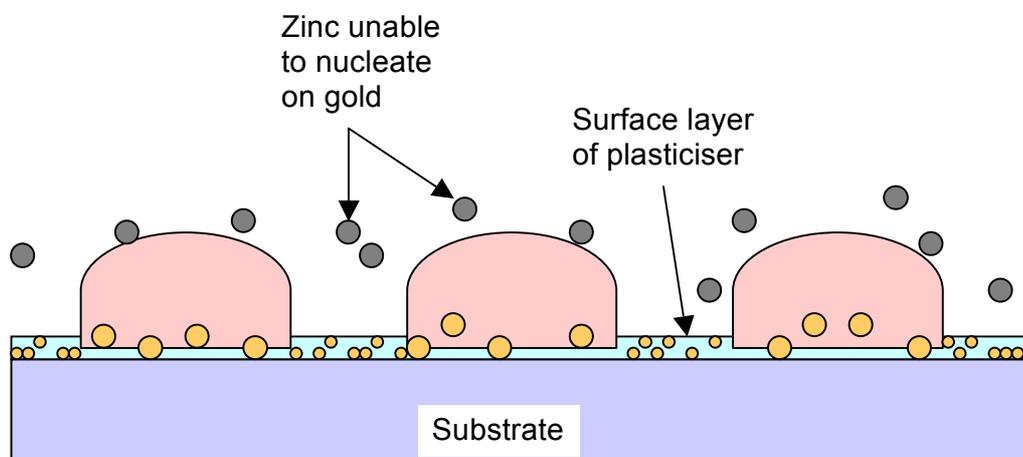
- 2.7 None of the theories above have been categorically proven, and in some cases reverse and normal development may be observed on the same substrate, although it is stated that this is most common for (if not exclusive to) LDPE substrates. The photograph below shows a 'reverse developed' mark on a polyethylene bag.



Photograph of a reverse developed mark on a polyethylene bag.

- 2.8 It is recognised that the gold/zinc VMD process does not work well (or at all) on substrates that are heavily plasticised (e.g. clingfilm, plasticised

PVC) or have surface release films or contamination. This is attributed to the fact that gold nuclei diffuse into the surface layer on the substrate as well as the fingerprint deposits, with the result that there are no nuclei on the surface of zinc to deposit on, as is illustrated schematically below.



Schematic diagram of no development, showing zinc unable to find gold nuclei on surface.

3. CAST processes

3.1 The process outlined in the *Manual of Fingerprint Development Techniques* [19] is essentially as follows:

- evaporate ~2mg of gold at a pressure of 3×10^{-4} mbar or lower;
- evaporate zinc at a pressure between $3-5 \times 10^{-4}$ mbar until a suitable coating is formed.

3.2 These steps can be repeated until the desired level of coating and fingerprint development has been obtained.

3.3 The reason for choosing these particular materials and conditions can be expanded as follows.

3.4 The role of gold in the VMD process is to act as the 'primer' for subsequent zinc deposition. Gold is not selective in that it will deposit across the entire surface of the exhibit, but the size and dispersion of the gold nuclei formed will be determined by the nature of the surface (chemistry, roughness, etc.). As outlined above, there is usually a sufficient difference between the nuclei formed in the regions of the fingerprint ridges and the background for the print to be delineated during subsequent zinc deposition. Gold is also used as the initial deposition metal because it is inert and does not react with fingerprint residues or atmospheric pollutants. The low deposition pressure is used so that gold can be deposited directly onto the surface without colliding with a significant number of molecules in the chamber, giving an even coating.

3.5 The role of zinc in the process is to delineate the fingerprint, primarily by the difference between the growth rate of zinc on the fingerprint ridges and the growth rate on the background. Zinc is highly effective for this purpose because it easily re-evaporates from the surface unless there is a suitable nucleation site present, thus the gold nuclei formed control the way in which zinc layers subsequently form. The sections above outline the different mechanisms by which differences in zinc growth rate can reveal fingerprints. The evaporation pressure used for zinc is higher than that for gold, and this is to allow the user more control over the zinc deposition process. Allowing more air into the chamber makes the deposition of zinc more uniform across the area of the exhibit. It was thought that the additional air molecules present in the chamber would reduce the kinetic energy of zinc atoms as they reach the surface and could increase development rate, but this has never been proven.

4. Critical issues

- 4.1 Sealed containers such as aerosol cans, sealed drink cans and bottles, batteries and items with sealed air pockets must not be treated using VMD because the expanding gases may cause the item to explode.
- 4.2 Articles to be treated by VMD must be dry and free of other residual liquids and solids.
- 4.3 During the zinc deposition stage of the gold/zinc VMD process it is essential for the operator to observe the development of the marks and to stop the process before any over-development occurs. The filament temperature and deposition time required to coat articles will vary according to the type of material and condition of the surface. For this reason multiple exhibits of different types should not be treated together.
- 4.4 Multiple deposition runs can be used to build up a coating if the initial run fails to develop any marks.

5. Application

- 5.1 Suitable surfaces: VMD has traditionally been recommended as the primary process for development of fingerprints on plastic bags and wrappings. Although still effective in this role it is no longer as effective as alternative processes, such as superglue and powder suspensions. The silver VMD process is one of the few techniques suitable for clingfilm. VMD is suitable for use on all types of non-porous surface, and is one of the more effective techniques on 'semi-porous' surfaces such as glossy magazines and wrapping paper, and the best process for the non-adhesive side of masking tapes.

- 5.2 The equipment used for VMD may vary according to manufacturer, but the essential elements of the system are the same. The equipment consists of a vacuum chamber capable of being pumped down to high levels of vacuum ($<3 \times 10^{-4}$ mbar), filaments for deposition of gold and zinc, and a viewing window so that the deposition of zinc can be monitored. The chamber may also contain a 'cold finger', chilled to low temperature to aid condensation of contaminants and to reduce pump down times. Articles to be coated are attached to the perimeter of the vacuum chamber, above the coating filaments. A typical system is illustrated below.



Typical vacuum metal deposition equipment.

- 5.3 The filaments used for deposition of gold and zinc are typically formed from thin sheets of molybdenum. The gold filament usually consists of a shallow dimple in a thin strip of molybdenum. This is because the quantity of gold used is very small ($\sim 2\text{--}3\text{mg}$), and it is important that all the gold reaches the substrate. If deeper containers are used, 'shadowing' may occur and not all regions of the article may be coated. Gold deposition takes place when the chamber has reached a pressure of 3×10^{-4} mbar or lower, and the current to the filament is increased until the filament reaches a yellow/white heat. Deposition of gold should be complete within ten seconds, but if any residue is observed on the filament as the current is reduced, the temperature should be increased again until all the gold has been evaporated.

- 5.4 Once gold deposition is completed, the pressure in the chamber is increased to $\sim 5 \times 10^{-4}$ mbar and the current to the zinc deposition filament(s) turned on. The reason for increasing the pressure in the chamber is to increase the uniformity of the coating produced. The zinc deposition filaments are larger and significantly deeper than the gold filament, and the quantity of zinc added is larger, typically 1g per run. The zinc used is in the form of foil, shot or powder. For zinc deposition, the current is increased until the filament glows a cherry red/dull orange colour. Once this occurs, the operator should observe the deposition process through the viewing window, ceasing deposition as soon as marks become visible on the substrate. After zinc deposition, the gold filament should be briefly heated to yellow/white heat to burn off any zinc contamination. The process is described in more detail elsewhere [19].
- 5.5 There is a great variability in the speed at which different substrates coat, and it may take over ten minutes to obtain a suitable coating on some types of material. In some cases it may be necessary to carry out multiple deposition runs in order to obtain satisfactory results, or to develop all the marks present. The presence of surface contamination, release agents or plasticisers may mean that it is not possible to obtain a zinc coating at all and in these circumstances the deposition of 30mg of silver using the same deposition conditions for gold may yield additional marks.
- 5.6 The VMD technique initially was adopted as an operational technique for the detection of latent prints on thin polyethylene items such as carrier bags and wrappings, and was shown to be superior to other processes developed subsequent to the initial comparison trials. Although the technique had originally been developed with the intention of being used to detect prints on fabrics, no identifiable prints were successfully obtained in operational trials and VMD is not currently (2011) recommended for operational use on this substrate.
- 5.7 VMD has now been used operationally for many years, and has been shown to be an effective technique for a wider range of materials than polyethylene. Recent results showing VMD to produce results on a range of substrates include a ticket coated with ferromagnetic ink, and on expanded polystyrene [20]. The use of the technique has also begun to increase in North America, and successful results have obtained from plastic bags, in some cases several years old and exposed to moisture [21].
- 5.8 The range of exhibits that have been successfully treated using VMD is extensive, and includes:
- plastic bags and packaging;
 - glass and plastic bottles;
 - firearms;
 - glossy card, photographic paper and magazine covers;

- clean leather items (including handbags and shoes);
- adhesive tapes (non-sticky side).

5.9 It is evident that there is much overlap between the types of article that can be treated with VMD and those that are treated using cyanoacrylate fuming. In many cases, the deciding factor as to which technique is to be used is whether the article has been wetted, because VMD remains effective on wetted items whereas cyanoacrylate fuming does not. In practice it is possible to use the two processes in sequence, and more marks may be detected in this way because the two processes work on different fingerprint constituents.

6. Alternative formulations and processes

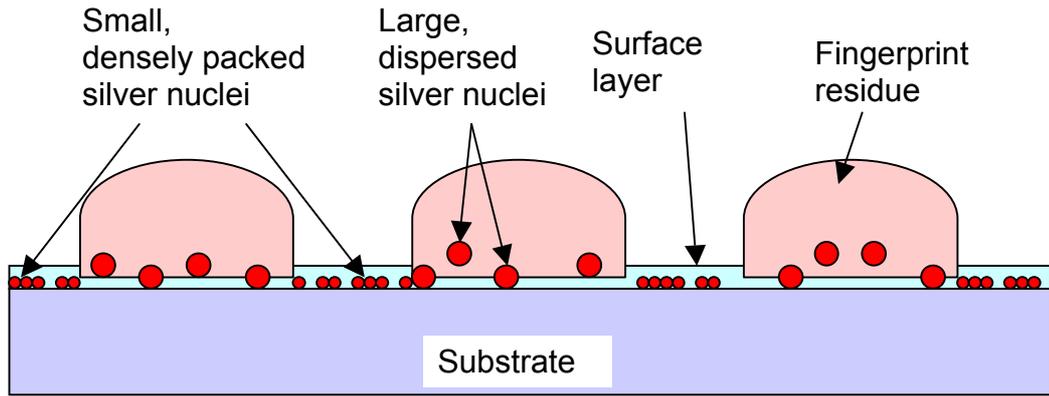
6.1 Several other materials have been investigated in the VMD process, including metal combinations, single metals, and organic materials. A summary of some of these is outlined below.

Metal 1	Metal 2	Comments
Gold	Cadmium	Initially, the gold/cadmium combination was selected as the optimum process, with cadmium giving better results than zinc when used as the second metal. It is also easier to produce coatings using cadmium. However, cadmium is very toxic and its use is no longer recommended on health and safety grounds.
Silver	Zinc	Silver can be used in place of gold as the initial deposition metal and limited evidence suggests that it this would have little effect on the effectiveness of the process. However, silver is more likely to interact with fingerprint constituents or atmospheric contaminants, and for this reason the more inert gold is preferred.
Silver	Cadmium	See comments for silver and cadmium above.
Copper	Zinc	Copper is potentially more reactive than silver or gold, and hence gold is preferred.
Copper	Cadmium	See comments for copper and cadmium above.
Lead	–	Of all the single metals investigated for fingerprint development in early studies (Hambley, 1972)[2], lead gave the best performance. However, lead is very toxic and its use is no longer recommended on health and safety grounds.
Zinc	–	Zinc is capable of developing fingerprints if used as a single metal, but re-evaporates

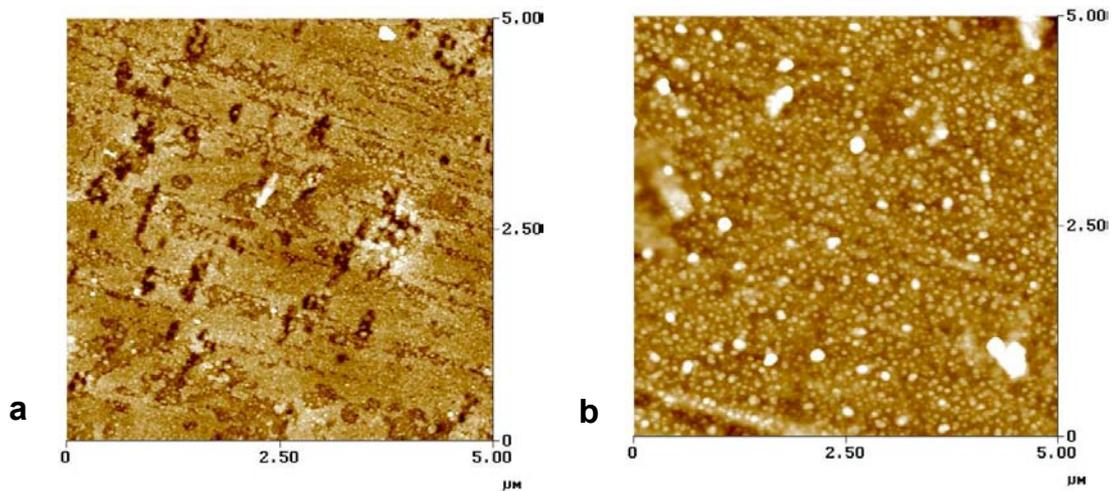
		easily from many surfaces and is best used in combinations.
Gold	–	Gold can be used as a single metal, and gives a blue background coloration with pink ridges. However, it has been found to be less sensitive than silver and copper and the gold/zinc combination when used this way.
Magnesium	–	Gives a silvery background, but less sensitive than most other single metals.
Copper	–	Gives a green/grey background coloration with pale yellow ridges. Effective on PVC-based clingfilm but less effective than silver on all other surfaces studied (Philipson and Bleay, 2007) [22]
Indium	–	Gives a pale brown background coloration with pale yellow ridges. Less effective than silver and marks difficult to see.
Tin	–	Gives a pale brown background coloration with pale yellow ridges. Less effective than silver and marks difficult to see.
Aluminium	–	Gives a silvery coating. Recently proposed as a more effective technique than gold/zinc on black plastic bags (Guraratne <i>et al.</i> , 2007)[23]. Ongoing research by CAST suggests no benefit over existing processes.
Silver	–	Identified as an alternative process to gold/zinc for plasticised materials (e.g. clingfilm) and materials with surface layers of contaminant (Philipson and Bleay, 2007)[22], now recommended for operational use by CAST. Can also be used sequentially after gold/zinc to fill in areas where zinc has deposited poorly. Further detail on the silver process is given below.

Summary table of alternative vacuum metal deposition processes.

6.2 The silver VMD technique is thought to work because silver, like gold, deposits uniformly across the surface. The nuclei formed vary in size and distribution between the fingerprint ridges and the background, giving a difference in colour between the two regions. This is shown schematically and as viewed by an atomic force microscope in the figures below.

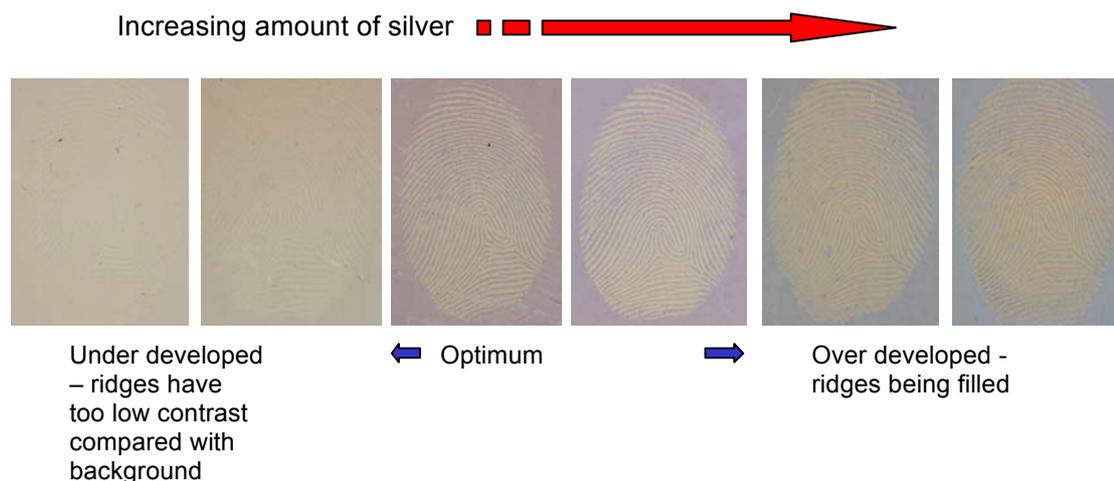


Schematic diagram of silver vacuum metal deposition on a plasticised surface, showing different sizes of silver nuclei in ridges and on surface.



Atomic force microscopy images of polyethylene bag after vacuum metal deposition, showing differences in silver nuclei size and density a) atomic force microscopy image of polyethylene surface, b) atomic force microscopy image of ridge region. Silver nuclei appear as light dots, and are smaller and very tightly packed on the polyethylene surface, and larger and more widely spaced in the fingerprint ridge.

- 6.3 The silver is deposited using the same conditions as for gold in the gold/zinc combination. The optimum amount of silver to use for most surfaces has been identified as 30mg. If less silver is used, development is too faint – if too much silver is used, ridges start to become filled in and detail can be lost, as seen in the sequence of images below.



Progression of colours developed using increasing amounts of silver in a single metal vacuum metal deposition process on polyethylene.

- 6.4 It is thought that copper works in a similar way, but the resultant colour of the film formed is different.
- 6.5 There has been recent interest in the aluminium deposition process [23], but trials at CAST have been unable to replicate the results in the literature. Most marks developed by this process are not easily visible and consequently are difficult to image. Comparisons of carrier bags cut in half and processed using aluminium and gold/zinc indicate that aluminium finds no marks from natural handling, only deliberately placed, 'groomed' marks.
- 6.6 Fluorescent, organic materials have also been deposited using the VMD process, most notably anthracene. Anthracene is less sensitive than most of the single metals and metal combinations outlined above and there are health and safety concerns regarding its use in this way. More recently, deposition of Rhodamine 6G in combination with an organic precursor has been investigated as a possible alternative to superglue fuming and dyeing [24]. The process was shown to develop fluorescent marks on surfaces, including metal, glass, plastic and thermal paper, but has not yet been developed further.

7. Post-treatments

- 7.1 A limited amount of research was carried out in the late-1970s on physical developer enhancement of VMD deposits on banknotes and metal images from banknotes transferred onto gelatine emulsions.

8. Validation and operational experience

8.1 The comparative effectiveness of VMD with other fingerprint development processes for the development of fingerprints on plastic (principally polyethylene) bags has been assessed in pseudo-operational and operational trials conducted by HOSDB. The principal results of these trials are reported below.

8.2 Laboratory trials

8.2.1 An initial laboratory trial conducted in 1978 [9] demonstrated that VMD typically developed between 23–27% useful marks on polythene bags compared with 7–10% for aluminium powdering, concluding that VMD was a superior process for this type of exhibit. This trial utilised planted marks deposited on plastic bags, results being obtained from over 1,000 deposited marks. The subsequent successful introduction of the technique into operational use meant that few other laboratory trials were conducted.

8.2.2 Laboratory trials were carried out when research was being conducted into deposition of alternative metals for development of marks on clingfilm [22]. These investigations compared the effectiveness of depositing silver and copper as single metals, on both polyvinylchloride (PVC)- and polyethylene (PE)-based clingfilms. Conventional gold/zinc VMD gave virtually no marks on both these types of clingfilm and was therefore omitted from the trial. Results for one-day-old and one-month-old marks are tabulated below. In the one-day-old experiment, 200 marks were analysed and in the one-month-old experiment, 240 were analysed.

Grade	Silver		Copper	
	PE	PVC	PE	PVC
3–4	10	20	0	24
2	4	10	0	11
1	36	10	10	10
0	0	10	40	5

One-day-old marks.

Grade	Silver		Copper	
	PE	PVC	PE	PVC
3–4	14	2	0	37*
2	28	24	3	5
1	1	12	40	0
0	17	22	16	18

* Many marks faint and difficult to image.

One-month-old marks.

Summary of comparative trials carried out on clingfilm using different vacuum metal deposition processes.

8.2.3 The results indicated that copper VMD was ineffective on PE-based clingfilm, but gave better results on PVC-based clingfilm than silver. Copper was only recommended for use if it was certain that the clingfilm found was PVC-based.

8.2.4 Comparative tests were also carried out between gold/zinc and silver VMD on two 'non-standard' clear packaging films, polyester terephthalate (PET) and cellophane, where gold/zinc VMD occasionally had problems with 'empty' prints or rapid fading of developed marks.

Grade	Gold/zinc		Silver	
	PET	Cellophane	PET	Cellophane
3-4	9	28	22	31
2	22	13	18	21
1	29*	19	10	8
0	0	0	0	0

* Many empty prints developed.

One-day-old marks.

Summary of comparative trials carried out on alternative clear packaging materials using different vacuum metal deposition processes.

8.2.5 It was shown that silver VMD offered an improvement over gold/zinc for development of marks on PET, and could also fill in ridge detail in regions where 'empty' prints developed. Although silver VMD performed well on cellophane, the developed marks faded very rapidly and there was no operational benefit in using the technique.

8.2.6 Finally, investigations were carried out into the use of gold and silver in combination, as opposed to silver as a single metal [25]. These showed no benefit in the use of gold-silver as opposed to silver and were not pursued further.

8.3 Pseudo-operational trials and operational experience

8.3.1 With the advent of the small particle reagent (SPR) process in 1979, an operational trial was conducted at Essex Police on plastic bag exhibits submitted to the fingerprint laboratory [26]. Each bag was cut in half, one-half being treated with VMD, the other with SPR. The results are summarised below.

Trial overview	
Number of polythene articles received	204
Number of cases received	57
Total number of fingerprints developed using VMD	117
Total number of fingerprints developed using SPR	61
Number of articles where VMD developed marks	36
Number of articles where SPR developed marks	20

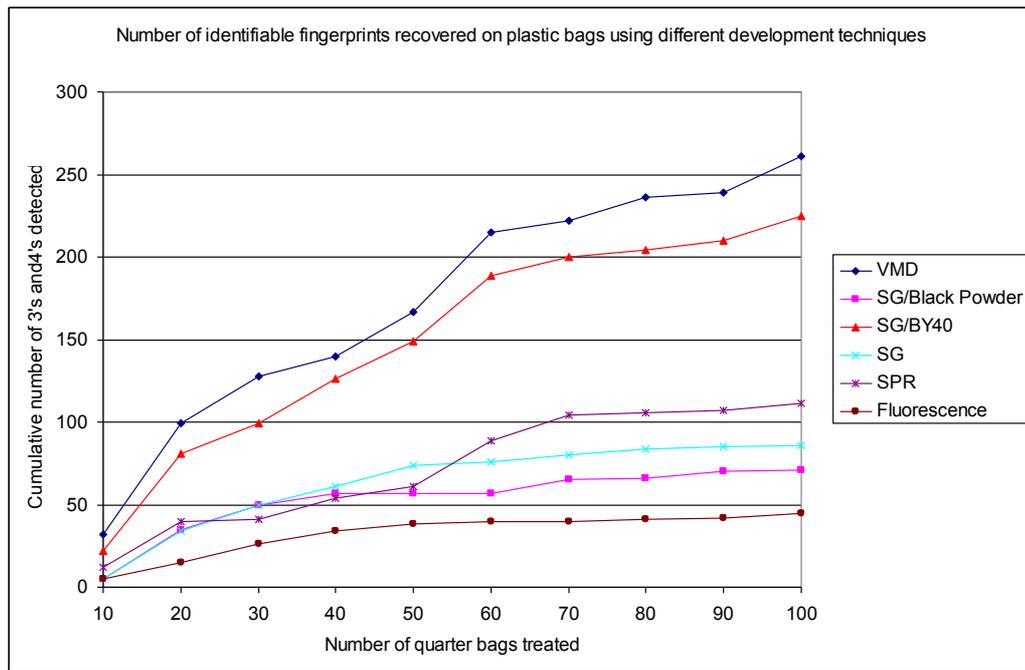
Process effectiveness comparison		
	Number of cases	Percentage from total number of cases
Cases with fingerprints only developed by VMD	13	23
Cases with fingerprints only developed by SPR	3	5
Cases where both VMD and SPR developed fingerprints	13	23
Cases where VMD developed fingerprints	26	46
Cases where SPR developed fingerprints	16	28
Total number of cases where VMD and SPR developed fingerprints	29	51

Early operational trial results comparing small particle reagent and vacuum metal deposition.

8.3.2 It was found that VMD was almost twice as effective as SPR on this type of exhibit. It was also observed that SPR could be used sequentially after VMD, but this was expected to be of only limited benefit.

8.3.3 The subject was revisited when an optimised superglue process became available in the mid-1980s, with a pseudo-operational trial being conducted between VMD, SPR, superglue and superglue followed by dyeing with basic yellow 40 and fluorescence examination. The trial was conducted by HOSDB on a large number of plastic bags using the same methodology as the study above, but not using operational casework.

8.3.4 The VMD process produced the largest number of identifiable fingerprints, producing approximately 12% more fingerprints than a combination of superglue, dyeing and fluorescence, and twice as many fingerprints as fluorescence alone [27]. Results of this exercise are shown below.



Results of pseudo-operational trial carried out on plastic packaging material in 1986.

8.3.5 More recently the composition of plastic bags has changed significantly, typically including more recycled material and observations from police forces using VMD on operational work indicated that the effectiveness had dropped off on this type of exhibit. As a consequence, the pseudo-operational trial above was repeated in 2009, comparing VMD with superglue and powder suspensions in a range of sequential processing scenarios. These studies are more fully reported in Chapter 3.7 Powder suspensions, and confirmed that VMD is no longer the most effective process for plastic bags, but instead should be used after superglue in a sequential processing route.

8.3.6 Operational trials involving silver have been more limited in extent because the process is only recommended as a secondary treatment after gold/zinc VMD. A small-scale study on clear cigarette wrappings (thought to be polypropylene) is summarised below, showing the number of wrappings yielding particular levels of ridge detail.

Result	Technique		
	Gold/zinc VMD	Silver VMD	Superglue fuming
Full print	1 (12.5%)	1 (5.6%)	1(12.5%)
Usable fragment	2(25%)	7(38.9%)	1(12.5%)
Unusable fragment	2(25%)	4(22.2%)	2(25%)
No print	3(37.5%)	6(33.3%)	4(50%)

Results of a short pseudo-operational trial on cigarette wrappers.

8.3.7 Silver VMD gives comparable results to gold/zinc VMD in this study, and better results than superglue. However, due to the limited sample size it is not possible to draw strong conclusions.

8.3.8 The process was trialled by some police forces on operational exhibits, using it after gold/zinc VMD where no development or patchy development was found. In these small-scale trials silver VMD was found to develop additional ridge detail in approximately 10% of cases.

9. References

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