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| **UNIVERSITY OF BRISTOL SCHOOL OF CHEMISTRY**  **RISK ASSESSMENT**  **This form must be completed by a competent assessor for any procedure/system of work before an attempt is made at carrying out the procedure/system of work. Please refer to the instructions for making a Risk Assessment in the** [**School of Chemistry Safety Manual**](http://www.chm.bris.ac.uk/safety/nfrass.htm) **(http://www.chm.bris.ac.uk/safety/nfrass.htm).** |
| **Name and Status of the Assessor: Hugo Dominguez-Andrade** (Senior Postdoc) **Date:** 09/11/22 |
| **Activity/procedure being assessed:**  Microwave plasma-assisted chemical vapour deposition of diamond |
| **Known or expected hazards associated with the activity:**   1. Electric shock, including from high voltage equipment 2. Leakage of microwave radiation from the reactor or waveguide (fire/burn hazard) 3. Overheating of microwave power supply from reflected power (fire/burn hazard) 4. Compressed gases & gas cylinders (gas escape; mishandling of gas cylinders) 5. Toxic/corrosive/explosive gases 6. Explosion hazard (in case of vacuum failure while reactor is operating) 7. Vacuum apparatus (implosion hazard) 8. Organic solvents (highly flammable) 9. Silicon/diamond (or similar) substrates (sharp) |
| **The risk of injury and its severity likely to arise from these hazards:**   1. The risk of injury from electric shock in normal operation is extremely low since hazardous high voltages are present only inside the microwave power supply and cannot be accessed without opening the equipment casing, which ordinarily is neither required nor appropriate. The potential severity is very high (death). 2. The risk of microwave leakage sufficient to cause injury is small since the reactor is designed and constructed in order to avoid this possibility, and an interlock system prevents the microwave power supply from being activated (or, if already activated, remaining on) unless the reactor is under vacuum and all important process parameters are within reasonable bounds. The severity of potential injury if the interlocks were to be defeated and the microwave generator operated without the reactor properly sealed is comparable to that from a domestic microwave oven, *i.e.* serious but not life-threatening burns on a portion of the body. 3. The risk of reflected power overheating the power supply is very low, as the system is optimised before being left to run and water cooling is present in the event of any power being reflected. Interlocks have been installed to ensure that the power supply is turned off and the gas inlet closed in the event of: reflected power rising above standard levels; reactor pressure rising above standard levels; H2 gas flow falling below safe levels; and water-cooling failure. 4. The risk of injury due to the escape of compressed gases is very low with properly fitted and serviced regulators, and with fittings properly made up and in good condition. The potential severity of this injury is low in the case of reasonably small releases of gas; however, releases of large volumes of inert gas at high pressure could cause serious physical injury (*e.g.* gas embolism) and the escape of methane or hydrogen could result in an explosion, with the potential to cause serious injury or death. All gas lines are maintained at the minimum practical pressure (typically 2030 psi), so that the only possible source of a high-pressure gas escape is from a cylinder or regulator, and these are situated in a separate room inside ventilated cabinets away from working areas; the most likely scenario is therefore the least severe case. The potential severity of injury due to transporting the cylinders is significant (*e.g*. broken limbs) as gas cylinders are heavy, unstable objects, but the risk of this injury is low if reasonable care is taken and the recommended handling procedures are followed. The risk and potential severity of explosion by reactor overpressure are both very low because the reactor is a closed metal vessel and designed not to seal against positive pressure, so the bursting force will be very small and well contained; at worst, a glass plate inside the reactor could be shattered. This is unlikely to occur, however, since all gas inlets are interlocked in order that gas flow will stop when the reactor reaches atmospheric pressure. 5. The risk of injury due to the escape of hydrogen (flammable; explosive), ammonia (corrosive; toxic), and diborane (toxic; pyrophoric) is low in the same sense that the risk of any gas escape is low, as stated above. Additionally, only small cylinders of ammonia and diborane (5% B2H6 in hydrogen) are used, and the fill pressure is lower than that used for standard cylinders, so that the maximum volume of gas potentially able to escape is inherently limited. The worst possible injury sustained from this is thus likely only to be moderately severe (pulmonary edema if inhaled at high concentrations) rather than extremely severe. However, this should still not be underestimated. The risk of a leak going unnoticed is very low, but unchecked, small leaks could result in the formation of an explosive atmosphere. In the case of hydrogen, this scenario is highly unlikely as a hydrogen detector is fitted inside the cylinder cabinet and this will sound an alarm in case of a potentially dangerous situation. 6. The risk of explosion due to vacuum failure and consequent ingress of air into the reactor while operating (i.e. containing hydrogen at 3000 K) is low in ordinary circumstances, but could be significant in case of unsafe working practices, operator negligence, or severe accident. An explosion of this sort could result in extremely serious bodily injury or death, since although the reactor is solidly constructed and incapable of sustaining significant overpressure, it is not intended to be explosion-proof, and the result of allowing an explosion to occur is not easily predictable. This is probably the most significant serious hazard associated with the activity. 7. The risk of injury from implosion is very limited because almost the entire apparatus is solidly constructed from stainless steel and aluminium. Small glass viewports are provided for observation, which may be able to be damaged through accident or negligence. However, the reactor volume is very small (< 1L), so an initial leak will result in a significant reduction in differential pressure and the kinetic energy of any fragments that may be able to escape the reactor will be such as is unlikely to cause any significant injury. 8. The risk of injury due to a solvent fire is low-to-medium: the risk of ignition is minimal, but large quantities of solvent are required for making up laser dye solutions, which increases the risk non-negligibly. The severity of this injury is likely to be low-to-medium in view of the primary application of solvents for cleaning samples, chamber fittings, and optics, which requires only very small quantities. The cleaning solvents usually employed are ethanol and acetone, so they pose only negligible toxic hazard. The potential severity of injury by methanol (sometimes used for cleaning optics) is greater due to its toxicity, but even smaller quantities are needed in this case, so that the overall impact is limited. 9. Substrates are normally handled with tweezers to avoid contaminating or damaging them; thus, the risk of cuts is low. Since the sharp objects in question are very small, the potential severity of a cut from them is inherently limited. |
| **Who is at risk?**  Research workers using the apparatus. There is a chance that other people nearby in the laboratory may be affected in the unlikely event that a major incident occurs. |
| **Measure to be taken to reduce the level of risk:**   1. All electrical items must be operated with no hazardous voltages accessible to users, and repair and maintenance of equipment must be performed by a competent person, and with electrical power completely disconnected wherever technically possible. Metal casings and other exposed parts of electrical apparatus must be bonded to earth ground to guard against possible dangerous faults. The general power supply must be connected and disconnected only when switched off, especially as there is no valid reason to do otherwise in any case. See also reference 1. 2. Interlocks must not be defeated and users must be made aware of the hazard potentially existing if the integrity of the reactor shielding is compromised. If modifications are made to the reactor, these must be carefully designed in order to prevent leakage of microwave radiation, and tested using a field strength meter to ensure that no hazard exists in practice. See also reference 2. 3. Ensure all interlocks are engaged before leaving the reactor unattended. Test interlocks on a regular basis to ensure their correct function. 4. Standard safe working practice must be followed for the handling and use of gas cylinders. In particular, special care must be taken when moving or transporting them (especially those containing hydrogen), and only persons physically capable of handling cylinders must attempt this procedure. Regulators must be checked for damage or improper operation each time a cylinder is replaced, since failure could lead to an overpressure condition in the downstream gas line. All fittings must be tested for leaks using bubble solution when first made up and whenever they are disconnected and reconnected. Users must be made aware of the hazard potentially arising from damaged or improperly handled compressed air lines, which are maintained at significantly higher pressure than other gas supplies. See also reference 3. 5. Cylinders of toxic or explosive gases must be kept inside securely closed, ventilated cylinder cabinets, preferably located in the cylinder store room. Lecture bottles, which hold small volumes of gas at low fill pressure and are brought to the experiment as required, must be firmly fixed in place and used with secure, leak-tested connections. Particularly toxic gases (such as carbon monoxide), for which even small leaks are potentially hazardous, must be used in combination with suitably placed gas-specific detectors to provide sufficient warning of the presence of the gas before dangerous concentrations are reached. Effluents pumped out of the reactor must be not be exhausted into the laboratory air, but rather directed via tubing into the foul air extraction system, which must be verified to operate at a sufficiently high inlet flow rate to rapidly dilute and remove hazardous gases. Harmful gaseous by-products of the plasma process must be given due consideration and dealt with appropriately, including a separate risk assessment if appropriate. See also references 4 and 5. 6. Extreme care must be taken not to disconnect any parts of the vacuum system while the reactor is in operation. All vacuum fittings must be securely made up, and of such a type as requires deliberate and concerted effort to disconnect. The quality of the vacuum seal must be checked each time the reactor is to be started, by ensuring that the achievable base pressure is sufficiently low as to exclude any hazardous air leak. No fittings may be adjusted with microwave power applied (except valves opened and closed as required for normal operation of the reactor), in case of possible mistake or accident. Care must be taken not to damage or disrupt any connections *e.g*. by rough handling, and no activities that could entail any risk of compromising the integrity of the reactor (including through misadventure) are to be permitted while it is operating. 7. No further measures deemed necessary, as the risk is negligible. However, see also reference 6. 8. Cleaning of apparatus and optics must be performed using a minimal quantity of solvent in a well-ventilated area, away from potential sources of ignition. Spills must be cleaned up promptly in order to avoid later accidental ignition. Bulk solvents must be stored in a suitable solvent cabinet, and wash bottles are to be of the vapour-venting type and stored appropriately. Protective gloves should preferably be worn to avoid skin irritation, and this is mandatory when the solvent used is methanol due to its established percutaneous toxicity. See also references 7 and 8. 9. No further measures deemed necessary. If a cut is sustained, it will be sufficient to wash with water (and optionally ethanol or isopropanol) and apply a sticking plaster. |
| **Training prerequisites:**  As required in the references. Training in the particular safety aspects of operating the apparatus is intrinsic in the training required in order to operate it in other respects, *i.e*. without damaging equipment and in order to obtain meaningful results. Untrained persons must not work on the experiment unsupervised. |
| **Level of risk remaining:**  Slight, barring unforeseeable accidents or negligent behaviour. |
| **Action to be taken in an emergency:**  IN CASE OF INJURY, perform first aid if trained or contact a designated first aider. (A current list is provided at the Porter’s Lodge). If necessary, also contact security on extension 112233 (0117 331 1223) to call for an ambulance.  IN CASE OF FIRE, use a carbon dioxide fire extinguisher, one of which is provided in the laboratory next to the apparatus; two more are located in the corridor near to the door from which one enters S111. A water fire extinguisher is also available in the corridor if required. If the fire is too large to deal with in this way, evacuate the affected area, raise the fire alarm, and contact security on extension 112233 (0117 331 1223) to call the fire brigade.  IN CASE OF GAS ESCAPE, evacuate the laboratory and any affected areas; if the gas is hazardous, inform anyone in the vicinity or raise the fire alarm as appropriate, evacuate the affected area, and contact security on extension 112233 (0117 331 1223) to call the fire brigade. |
| **References:**  The following constitute an integral part of this risk assessment and must be reviewed along with the above.  Standard School of Chemistry risk assessments (see http://www.chm.bris.ac.uk/safety/compform.htm**)**:   1. Use of standard electrical equipment 2. Use of high-power Microwave and Radiofrequency power supplies 3. Transport and Use of Compressed Gas Cylinders 4. Use of Flammable, Explosive and Toxic Gases 5. Use of Diborane Gas (if applicable) 6. Use of Reduced Pressure or Vacuum 7. Transport, Storage and Use of Solvents and other Flammable Liquids 8. Disposal of Waste solvents 9. Manual Handling 10. Visual Display Equipment |
| **Signature of Assessor: Supervisor’s signature:**  Hugo Dominguez Andrade Neil Fox |