ANTI-FOULING COATING TECHNOLOGIES : A CRITICAL REVIEW AND DEVELOPMENT ROADMAP

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ABSTRACT

The deposition process of fouling on thermal surfaces is a complex phenomenon, influenced by several, interacting parameters: operating conditions; feed composition; geometry of heat exchangers; surface topology and properties (e.g. roughness, wettability). One possible fouling mitigation strategy is to limit or inhibit the fouling attachment on the heat transfer surfaces via surface modification. In the open literature, several methodologies, either vacuum or non-vacuum, have been shown to produce coatings that exhibit remarkable anti-fouling effects. Unfortunately, there are still unresolved issues that are limiting the effectiveness of the solutions available, including limited lifetime; only thick coatings show greater durability adding a non-negligible thermal resistance; retrofitting *in situ* of existing equipment is cumbersome, uneconomic, or simply not possible. This work aims at reviewing the main advantages and limitations of the most recent coating technologies, highlighting the current trends and envisaged future directions. A roadmap for the development of novel nano-based surface functionalization techniques capable of overcoming the current limitations of existing coatings methods is also proposed.

INTRODUCTION

Formation of organic and inorganic deposits associated with fluid heating processes in the heat transfer systems is one of the most complex operational challenges in the process industry [1-4]. Fouling is one of the major factors hindering energy recovery in process plants, and is often associated with large additional costs of maintenance, operations, production and the environmental [3,5].

From an operational point of view, when fouling deposits form on heat transfer surfaces, the heat transfer rate is impaired and the pressure drops are increased, thus the efficiency of the heat transfer system is reduced and production is affected [3, 6-9].

Energy consumption in industrial processes is massive, estimated to account for 37% (157 EJ) of total global final energy use worldwide [10]. For example, fossil fuels are still extensively used to produce heat needed to generate steam for the production of electrical power. In these processes, a large fraction of the heat released from the fuel is recovered with the use of various heat exchangers that transfer the heat to a cold utility (cooling water). Under the prevailing conditions of operation there is ample opportunity for heat transfer surfaces to become fouled with related reduction in the efficiency of energy utilization. In other processes the primary fuels, coal, oil or natural gas but even solar energy (*i.e.* concentrated solar pawer plants or solar thermal systems) are used for process stream heating. Reduced efficiency of the heat exchangers due to fouling in these systems, produces an increase in fuel consumption or, if renewable sources are used, it is associated with a less efficient use of the unit of energy, with repercussions not only in cost but also in the conservation/use of the world's energy fossil/renewable resources. The necessary use of additional fossil fuels to compensate for the shortfall in energy recovered due to fouling, also has an impact on the environment. The increased carbon dioxide produced during combustion adds to the "global warming" effect. In the current climate of great environmental concerns, conservation of limited resources and clean air, this stresses further the role that fouling mitigation has to play at a global scale if the ambitious net zero targets set by world-wide governments and private companies are to be met.

While reduced heat transfer efficiency is a major and well recognized impact, fouling deposition may also add pressure drop problems to the system. The presence of the fouling deposits restricts the cross sectional area available for the fluid to flow thus resulting in increased pressure drop [3]. In extreme examples, this increase in pressure drops cannot be overcome by the hydraulic circuit and production needs to be stopped with consequent large economic penalties.

Finally, the repeated and persistent need to shut down the plant to clean heat exchangers, has important safety implications. Very often to stop and open heat exchangers requires the use of cranes and large trucks to move heavy bundles to wash pads. The cleaning actions themselves could be very dangerous, particularly when manual hydro-blasting

– the use of 1,000 bar water jets to remove fouling deposits– is used.

Mitigation of the fouling impact can be achieved by using various approaches, including:

- Selecting a suitable heat exchanger geometry (e.g. using tube inserts, special heat exchanger constructions, alternative shell-side geometries such as helical baffles [11], Twisted Tube® [12], EMbaffles® [13], rod baffles)
- Manipulating fluid chemistry (e.g. injecting chemicals in the process fluid, physical water conditioning)
- Engineering the surface properties (e.g. using electrophoresis or using coatings)
- Using digital technology to devise and implement a cleaning schedule that optimizes the cleaning actions (*i.e.* when to clean, which heat exchanger to clean and how to clean it and for how long)

Each of the above has its pros and cons but the applicability and effectiveness of all these technologies are highly dependent on a number of factors (which results in conflicting reports on their success).

For instance, the production of water treatments to prevent is a US$ 7.3 billion p.a. industry. Of these chemicals, 40% are purchased for control of the scale in cooling towers, boilers and other heat transfer equipment [14]. The various chemicals added to the water fall into three categories, *i.e.* control of biological growth, prevention of scale formation, and corrosion inhibition. Careful choice of treatment programs may indeed help addressing the problem but the use of chemicals often produces problems for the environment as they are discharged in the air, water streams and reservoirs and are buried in landfills. An environmentally-friendly approach to overcome these fouling problems should avoid as much as possible the use of chemicals harmful to the environment.

This paper presents a critical review of selected coating techniques that can be used for antifouling purposes as reported in the open literature. Proprietary technologies that may be available commercially are excluded from the scope of the review. It should be highlighted that the aim of the paper is not to review the coatings themselves, but rather highlighting advantages and limitations of the techniques used to produce them. The interested reader can find comprehensive review of existing coatings in the open literature or from other sources [15]. A roadmap for future development of novel nano-based surface functionalization techniques capable of overcoming the current limitations of existing coatings is also proposed.

COATING TECHNOLOGIES

Coating technology has a long history of academic and industrial research [16] and there are several coating technologies available in the market[17-19]. Each of these technologies has its own advantages and limitations which are usually related to the specific process involved [16].

The different coating technologies can be compared on the basis of different key performance indicators, such as:

1. Costs: this includes the cost of the equipment and the cost of the application of the coating to the surface (material, labour etc)
2. Size of the surface that can be coated
3. Geometry (*i.e.* shape) and complexity of the surface that can be coated
4. Material capability
5. Deposition temperature range: max and min substrate temperature that can be reached during the coating deposition. This affects the choice of the materials used for the coating.
6. Deposition rate (*i.e.* how long it takes to apply the coating to the surface)
7. Uniformity of the coating
8. *In situ* deployment: ability to retrofit existing equipment *in situ*
9. Coating wastage
10. Safety and environmental compatibility.

Table 1 compares the current characteristics of the most common coating technologies.

Existing vacuum processes, such as Physical Vapor Deposition (PVD) (e.g., e-beam deposition, sputtering), Chemical Vapor Deposition (CVD), Plasma processes, produce stable, controllable, and reproducible coatings but need large capital investments while the sample size and complexity are limited by the dimension of the vacuum chamber. Moreover, the large amount of energy involved, and the required deposition temperatures limit their application to temperature insensitive materials [20, 21].

Novel non-vacuum processes, such as atmospheric pressure plasma, allow coverage of temperature sensitive materials but the type of coating materials is limited [16]. Wet-chemical methods (dip coating, spin-coating or spray-coating) are simple to apply, but often require hazardous and toxic precursors and solvents [20].

Among the coating methods reported in Table 1, only the dip coating allows for *in situ* deposition. However, similarly to all wet chemical processes, it suffers of slow deposition rates and large wastage of the coating material. It should be noted that the evaluation reported in the Table 1 is based on direct experience of the authors and/or literature information. Some characteristics (e.g. longevity or adhesion strength) are related to the single coating and difficult to come by in the open literature. Moreover, it should be borne in mind that the success of the coating strongly depends on system it has been applied to, the operating conditions it has been subjected etc.

Table 1. Comparison between the current coating technologies [16-19]

KPI	**Description**	**e-beam (PVD)**	**Sputtering (PVD)**	**CVD**	**Plasma Enhanced CVD**	**Dip coating**	**Spin-coating**	**Spray-coating**
	Vacuum	Yes	Yes	Yes	Yes/No	No	No	No
1	**System cost**	High The main cost is for installation (capital costs)				Low cost on the equipment. The main cost is on the chemicals		
2	**Size**	Limited by the vacuum chamber			Limited	None	Limited	Unlimited
3	**Shape complexity**	One side - 2D – step coverage issue				3D	1D	3D
4	**Materials capability**	Simple compositions	Nearly unlimited	Low	No vacuum limited	Limited by precursors and required curing temperatures		
5	Deposition temperature	Medium - High	Medium - High	High	Low	Low	Low	Low
6	**Deposition rate**	Moderate	Long	Long	Long	slow rate	slow rate	slow rate
7	**Uniformity**	Variable	Fair to good	Good	Fair – good	Fair - good	good	Variable
8	***In situ* -Multiple**	No	No	No	No	Yes	No	No
9	**Coating wastage**	-	-	-	-	High	High	Moderate
10	**Safety and environment**	Safe but highly energy consuming				Use of toxic, hazardous precursors and solvents		

ANTI-FOULING COATINGS

Surface modification methods showed remarkable effects on anti-fouling. However, they also present a few major intrinsic limitations, which are hindering their effective deployment in the process industry. In particular, they show a relatively average limited lifetime, which is in the order of weeks. Coatings that present a longer lifetime are usually very thick and, thus they lead to non-negligible additional thermal resistance that is undesirable in heat transfer applications. Finally, none of the coating methods reviewed allows for the retrofitting of large equipment.

As a result of the above, the question becomes it is it possible to overcome these issues and, if so, what is the most suitable methodology to use?

Several different novel methodologies have been recently proposed in the open literature and most of those involved innovative deposition methods based on nanotechnologies. In fact, the use nanocoatings for surface functionalization allows to produce ultra-thin layers that help ensuring the additional thermal resistance is kept to a negligible level. Anti-fouling surface can be either super-hydrophobic or super-hydrophilic.

Yin *et al.* [22] developed a fluorine-free anti-fouling Ni_3S_2 coating on 304 Stainless steel. According to Figure 1, the method proposed consists of a first electrodeposition of a pure nickel thin coating. In a second step, the pre-coated sample underwent to a solvothermal 2.92 mmol TAA, 0.65 mmol $NiSO_4$, 45 mmol NaOH and 2.74 mmol CTAB were dissolved into 60 mL ethanol solution (volume ratio 1: 1) and mixed well by stirring for 10 min.

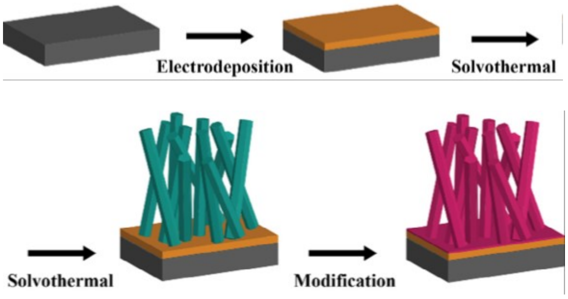


Fig. 1. Deposition method proposed by Yin et al. [22]

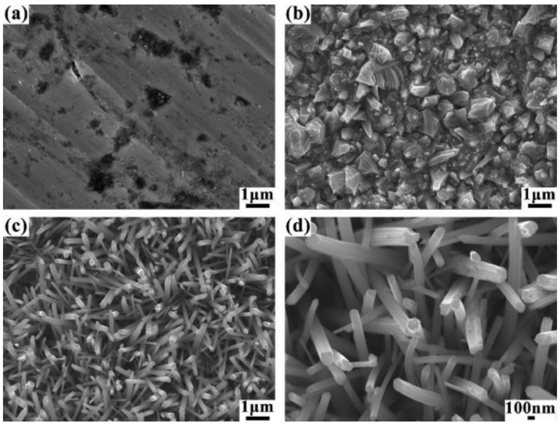


Fig. 2. Fluorine-free anti-fouling Ni_3S_2 coating on 304 Stainless stee by Yin et al. [22]

In the following step, the sample and mixture were transferred into a Teflon-lined stainless steel autoclave. The sealed autoclave was kept at 170 °C for 6 h. The final sample was rinsed with abundant ethanol and dried at 60 °C for 2 h.

Fig. 2 reports the morphology of the super-hydrophobic samples obtained with the described procedure. In particular, Fig. 2a presents the pristine stainless steel while Fig. 2b the electrodeposited Ni coating. After solvothermal reaction, the surface was covered with black coating and dense 1D nanorods (50–250 nm) can be seen as shown in Fig. 2c. From Fig. 2d, the hierarchical structure of stepped protrusions existed on the nanorod surfaces for constructing superhydrophobic surface can be observed.

Moreover, the coating developed by Yin *et al.* [22] maintains excellent repellence to water (Fig.3) even when tested in harsh conditions via long-term ethanol immersion or heating treatment at 300 °C. Additional O_2 plasma etching can cause this super-hydrophobic coating to become super-hydrophobic, the super-hydrophobicity will be regained after heating treatment (Fig.4).

Holberg *et al.* [23] proposed a novel biocide-free fouling-release coatings by dispersing a polydimethyl siloxane (silicone, PDMS)-polyethylene glycol (PEG) copolymer in a PDMS coating. The authors compared the anti-fouling and fouling release performance of the proposed coating against those exhibited by known coatings including commercial fouling release for marine vessels. The tests were run in laboratory (contact angle, pseudo barnacle test and bacteria culture test applying *Pseudomonas aeruginosa*) and by application on fresh water-cooled surface condensers mimicking conditions of thermal power plants. The proposed solution reduced fouling growth and adhesion in laboratory tests by more than a factor of ten compared to steel. However, when exposed to freshwater, the commercial fouling release coating performed better than the developed one and is a promising candidate for applications on surface condensers of thermal power plants as it reduces fouling at a flow rate of just 1.6 m/s. As shown in Fig. 5, the coating resisted 8 weeks when exposed to fresh water at 40 °C, at 1.6 m/s flow in test run applying water from the river Seine in Chatou, France.

A different approach was followed by Wang *et al.* [24] to prepare an anti-fouling coating for metal pipeline for geothermal application. In fact, a novel anti-fouling epoxy-silicone composite coating containing Ni–Cu–Al alloy powder was fabricated based on the basic principles of galvanic corrosion.

Fig. 3. Super-hydrophobicity of the fluorine-free anti-fouling Ni_3S_2 coating on 304 Stainless steel by Yin *et al.* [22]

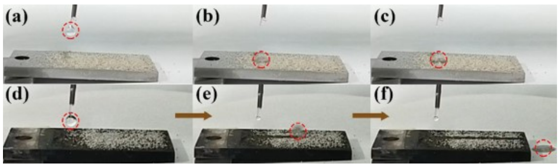


Fig. 4. Self-cleaning process on the surface of (top) steel substrate and (bottom) superhydrophobic Ni_3S_2 coating l by Yin *et al.* [22]

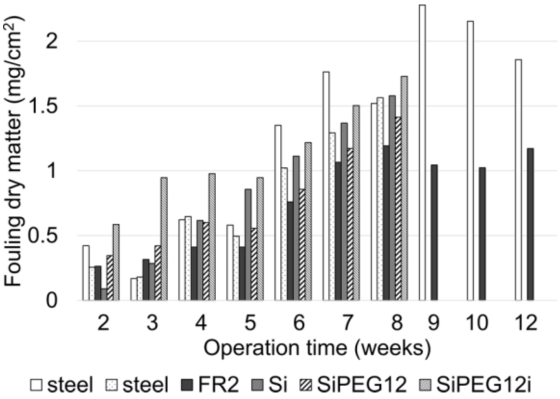


Fig. 5 Freshwater test of different anti-fouling coatings. Holberg *et al.* [23]

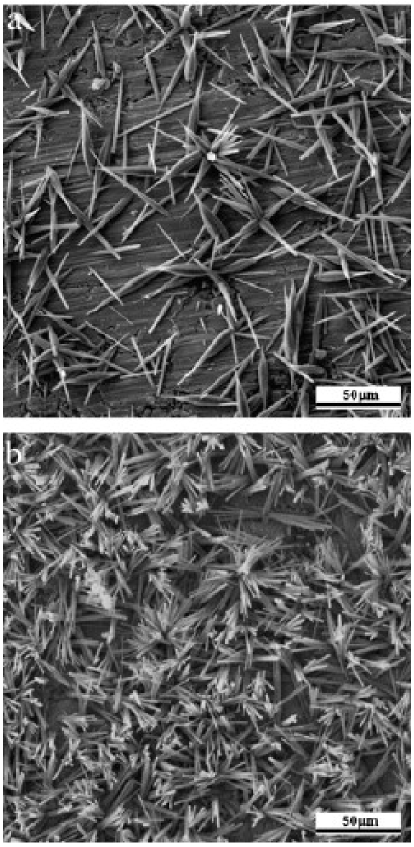


Fig. 6 SEM images of specimens immersed in different test vessel for 72 h. (a) coated; (b) uncoated. Wang et al. [24]

The process involved a flake aluminum blended copper and nickel powder with a constant copper/nickel/aluminum ratio of 1/1/0.1 by weight that was prepared in a rotary blender. The stainless steel specimens were treated and brushed with the slurry coatings. The slurry-wetted stainless steel specimens were preheated in an air oven at 70 °C for 20 min, then heated at 180 °C for 60 min followed by cooling to room temperature in air environment.

Galvanic corrosion reactions cause Ni_2+, Cu_2+ and Al_3+ ions cumulate in the vicinity of composite coating. Moreover, being driven by the ion concentration gradient, there would be an amount of Ni_2+, Cu_2+ and Al_3+ ions in bulk solution. Those metal ions intensively inhibit the nucleation and crystal growth rate of $CaCO_3$ fouling precipitating on the surface of composite coating and anti-fouling performance of composite coating would be improved. In order to evaluate the anti-fouling performance of the proposed coating, both coated and uncoated stainless-steel specimens were immersed in different vessels containing 500 ml simulated geothermal water without stirring at 50 °C. During the first test the samples remained immersed for 72h, then they were analyzed. As shown in Fig. 6, after the immersion test in the simulated geothermal water at 50 °C for 72 h, compared with stainless steel and epoxy-silicone resin coating, only a small amount of fouling was observed on the surface of composite coating and the composite coating showed a good anti-fouling performance.

Ahn *et al.* [25] developed a novel method to prepare anti-fouling coating to improve the performance of chevron plate heat exchangers. The coating process consists in two steps: the first one is used to modify the stainless steel surface to produce micro/nanoscale holes using an electrical etching technique. Then, during the second step the modified surface is coated with polymer (polymethyl methacrylate; PMMA) and hexagonal boron nitride (BN) particles to obtain the hydrophobic and superhydrophobic wetting characteristics. A schematic of the electrochemical etching setup used in the first step is reported in Fig. 7.

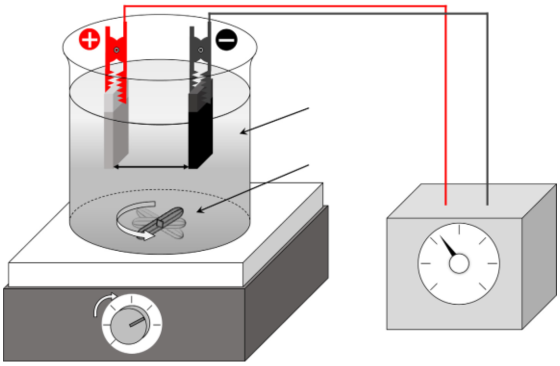


Fig. 7 Electrochemical etching setup. Ahn et al. [25]

Aqueous HCl (36%) and HNO_3 (69%) solutions were prepared and mixed in a 3:1 vol ratio to make a dilute 5.2% aqua regia solution. The solution was stirred continuously between its preparation and the surface etching. The stainless-steel specimen was placed parallel to a carbon plate at a distance of 3 cm. Two plates were immersed in dilute aqua regia solution and a constant electric potential was applied for the electrochemical etching. The etching period was 10 min with a DC potential of 7 V. The specimens were rinsed in deionized water immediately after etching and baked at 40 °C for 1 h in an oven. The additional PMMA polymer and BN coatings were deposited on the modified specimens using the spray coating method. The PMMA polymer was prepared with 50% vol. in anisole (methyl phenyl ether) solution sprayed onto the modified specimen for 2 s. The curing was done by drying in a convection oven at 100 °C. The BN coating was fabricated from BN particles (hexagonal BN with size <5 lm), which were made into a uniform coating by spraying onto the modified specimen for 2 s.

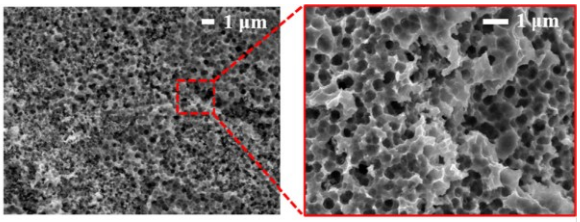


Fig. 8 SEM image of etched specimen. Ahn *et al.* [25]

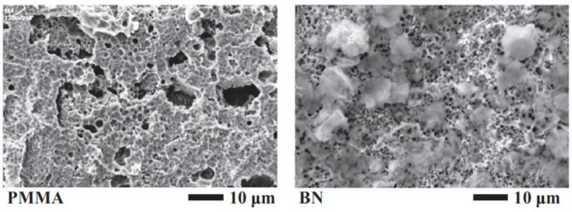


Fig. 9 SEM image of coated plates. Ahn *et al.* [25]

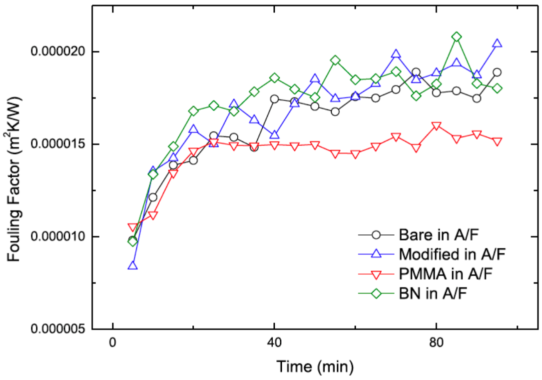


Fig. 10 Fouling factor for the tested samples Ahn *et al.* [25]

The BN-coated specimens were dried for 10–20 min at room temperature, and annealed at 300 °C for 1 h to burn away the solvent from the sprayed BN solution. Fig. 9 shows two SEM images of the coated samples.

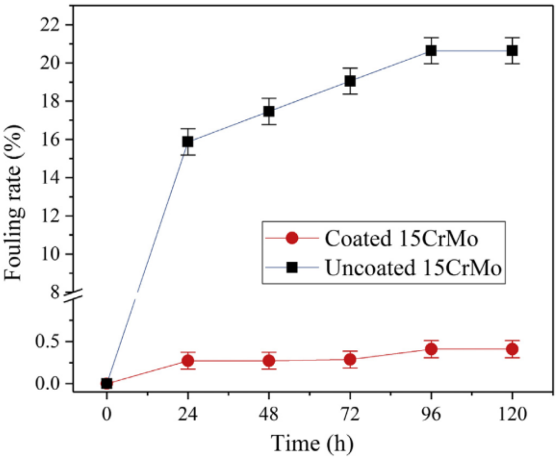


Fig. 11 Surface fouling rate curves of the uncoated and coated steel sheet specimens exposed to sodium sulfate at 550 °C as a function of the fouling time. Each fouling cycle comprised of 24 h. Wang et al. [26].

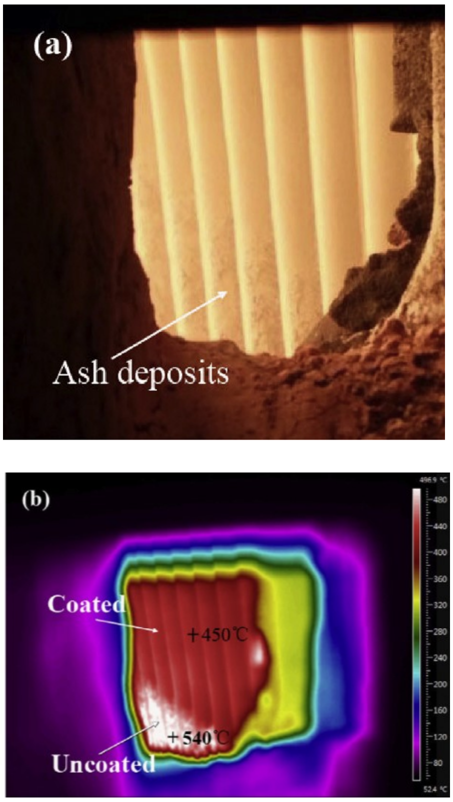


Fig. 12 Photography (a) and IR thermography image (b) of the ceramic coating used in real-life boiler system. Wang et al. [26].

The authors [25] performed accelerated tests to evaluate the performance of the modified/coated samples; as shown in Fig. 10, the fouling pattern of the fouling acceleration experiments in $CaCO_3$ solution presented the asymptotic pattern which would be balanced with the deposition and the removal of precipitated $CaCO_3$ within 2 h. The PMMA coating exhibited the best performance.

Finally, Wang et al. [26] proposed a mitigation coating for fouling problem in boilers fired with high-sodium coal (HSC). In fact, Wang et al. [26] developed a composite ceramic coating the slurry method that was applied on 15CrMo steel. After sintering, the composite ceramic coating had a dense structure and was well bonded to the substrate forming a metallurgical damascene structure. As shown in Fig 11, the authors found that fouling and thermal shock resistance of this ceramic coating to sodium sulfate was excellent. Fig. 12 shows the significant difference in the fouling resistance between the uncoated and coated areas of the heating surface. The coated surfaces were relatively clean, whereas ash deposits visibly covered the uncoated surfaces. The IR thermography image revealed that the temperature of the pipewall in the uncoated area was higher by 90°C compared to the coated area

A ROADMAP TOWARDS THE FUTURE ANTI-FOULING COATINGS

Following the literature review above, it is clear that, while there are several promising methods and successful applications, there is no ultimate coating technique that is inexpensive (so that it can be applied at large scale), that can easily be applied to existing equipment *in situ* and that produce coatings with low thermal resistance which are long-lasting in harsh industrial services.

To overcome these limitations, a completely different approach to coating technology is needed. In particular, the research should focus on the development of novel methodologies to enable:

- High thermal performance of the coating (whether via high thermal conductivity and/or via very small thickness)
- Cost reduction via a drastic simplification of the deposition procedure and the use of inexpensive and commonly available materials;
- Flexible coatings and materials selection procedures to coat a variety of applications and complex geometries;
- *In situ* deposition without expensive machinery to deploy the coatings in existing equipment and rejuvenate the surface after cleaning, if needed;
- Simple and effective scale-up to large number of units.

The development roadmap proposed includes the following steps:

1. Identification of the desired qualities of the coating technique as well as the protocols and the KPIs for the evaluation of the resulting coatings.
2. Selection of the most promising techniques that can deliver said qualities.
3. Small scale demonstration of the heat transfer characteristics in single phase and pool boiling
4. Pilot plant performance tests with realistic heat exchanger sections (e.g. tubular and plate) and operating conditions for selected applications

5. Pilot plant longevity test
6. Identification of a technology coating partner to scale up the procedure at plant level
7. Pilot test with operating company
8. Full commercial roll-out

Having completed steps one, two and three a novel deposition method based on nano-colloidal suspension has been developed at the Nano Heat Transfer lab of the University of Padova [27] and demonstrated to show many favorable characteristics. The method is now under development in collaboration with Hexxcell Ltd. to design effective coatings for targeted applications, particularity with respect to fouling.

This novel method enables the deposition of ultra-thin nanocoatings for surface functionalization which is not limited to anti-fouling and can be tailored to other applications too (e.g. enhanced boiling, condensation etc). The process is run at atmospheric pressure with no restriction in size of the sample to be coated; coatings on complex 3D structures (e.g. heat exchanger fins) can be easily obtained. The process also allows the deposition of temperature sensitive materials with tunable deposition rates.

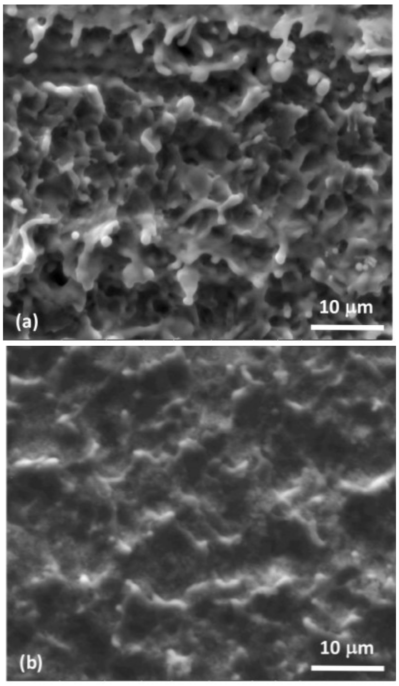


Fig. 13 SEM images of the sample (a) before and (b) after nanoparticle deposition at 5000× magnification. Mancin et al. [27].

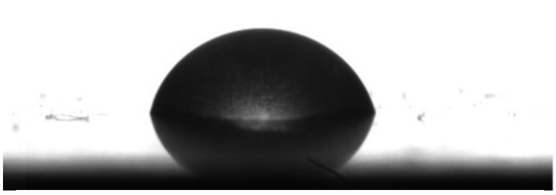
One of the main advantages of this method is the ability to perform *in-situ* deposition opening interesting possibilities for the retrofitting of large equipment. Finally, this deposition method does not use any toxic precursor or primer and it is completely environmentally friendly.

As an example of coatings already obtained, Fig. 13 shows SEM images of the copper surface before and after Cu nanoparticle deposition while Fig. 14 highlights the effects of the ultra-thin nanocoating on the wettability of the surface. Preliminary results obtained in terms of heat transfer characteristics, ease of deployment, deposition rate and attachment to the surface are very promising. However, additional tests are needed to confirm the suitability of this novel coating technology in terms of its longevity and its ability to produce effective anti-fouling coatings.

CONCLUSIONS

The paper reviewed promising coating technologies for antifouling applications under study in several international laboratories. From the literature review it emerged that, while there are several methods suitable to produce anti-fouling coatings, there is no an ultimate coating methodology that is inexpensive (so that it can be applied at scale) and that can be easily be applied to existing equipment *in situ*. Qualities of desired coating technique have been highlighted and a roadmap to obtain it has been outlined.

Static Contact Angle = 73.4° ± 3.7°



Static Contact Angle = 134.0 ± 3.4°

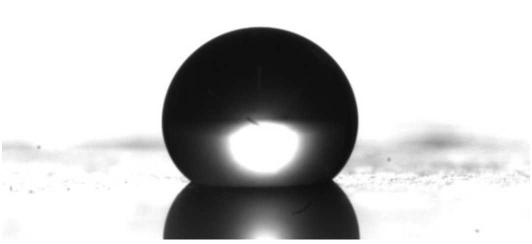


Fig. 14 Static contact angle measurements on the sample (a) before and (b) after the nanoparticle coating. Mancin et al. [27].

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