The effect of annealing on the mechanical properties of iron: stainless steel composistes

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The Effect of Annealing on the Mechanical Properties of Iron - Stainless Steel composites

Rajib Barua[1], Huseyin Aydin[2], David Frost[1], Rosaire Mongrain[1], Phuong Vo[3] and Stephen Yue[2]
[1]Department of Mechanical Engineering, McGill University, 817 Sherbrooke Street West, Montreal, Quebec, H3A 0C3, Canada; [2]Department of Mining and Materials Engineering, McGill University, 3610 University Street, Montreal, Quebec, H3A 2B2; Canada; [3]NRC Boucherville, Quebec.

Stainless steel (316L) powder is mixed with commercially pure iron (CP Fe) (by weight of CP Fe in the range between 20% and 80%) and cold sprayed onto a steel substrate to produce a metal composite designed primarily to exhibit controlled properties. For these composites, porosity is very low, but annealing between 600°C to 1100°C for an hour reduces porosity; the lowest porosity was exhibited by the 50-50 composite. Annealing also 'sinters' the interparticle interfaces, leading to vastly improved fracture properties. The fully annealed single component 316L material exhibits a much higher strength compared to the fully annealed CP Fe specimen, but the addition of only 20% 316L to CP Fe leads to a composite with the same fully annealed strength level as that of the 316L.

1 Introduction

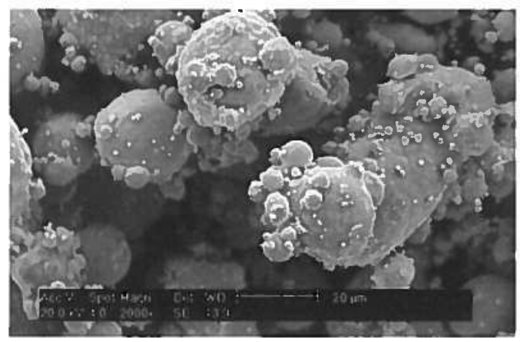
Composite metal-metal materials produced by spraying powders of two or more different metal powders can provide desirable multifunctional properties [1]. Some metals and alloys been targeted for mixed powder applications by using cold spray [1, 2-4] for biomedical applications. Hydroxyapatite/ Ti mixed powders have been sprayed to fabricate load bearing surgical implants with less cytotoxicity and better mechanical properties [5]. Mixed coatings for metallic biodegradable coating were based on cobalt-chromium L605 alloy mixed with stainless steel 316L in order to explore the possibility of micro-galvanic corrosion [6].

In this work, 'commercial purity' (CP) Fe is mixed with 316L stainless steel to nominally produce composites with CP Fe compositions of 20%, 50% and 80wt% by cold spraying onto steel substrates. The primary concern was to control the corrosion characteristics of the composite, but also of great importance are the mechanical properties of the coatings. This paper describes the mechanical properties of these composites in the as sprayed and annealed conditions.

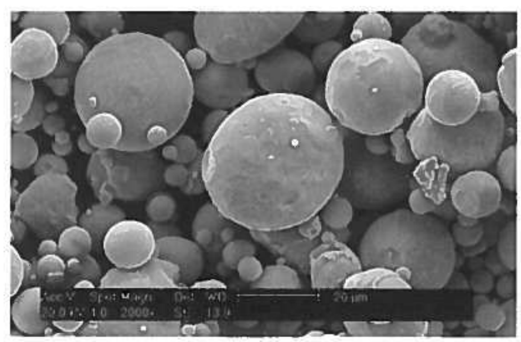
2 Materials and Methods

Feedstock powders were purchased from Sandvik Osprey Limited, UK. The 316L stainless steel contained about 16 wt% Cr, 11 wt % Ni and 0.014 wt % C. The 'commercial purity' (CP) Fe had about 0.4 wt % Mn and 0.03 wt% C. The average powder diameters were about 44 μm and 23 μm for the 316L and CP Fe, respectively. Both powders were basically spherical (Fig. 1) but the 316L had very fine particles coating the 44 μm particles. The microhardness values of CP Fe and 316L powders are about 290 and 230 $Hv_{0.01}$, respectively, which are much higher than the bulk values of 150 and 155 Hv for Fe and 316L, respectively [7,8], and may be due to the presence of martensite.

The composites were fabricated using a KINETICS® 4000 cold spray system (Sulzer Metco, Westbury, NY) with nitrogen as the propellant gas and an MOC24 nozzle at the McGill – NRC Cold Spray facility housed in the National Research Center (NRC), Boucherville, QC. Mixed powders were prepared by using either a rolling mixer without balls or a rotating tumbler with balls for an hour. Mild steel 1020CR substrates were blasted with 24 grit alumina before spraying to increase adhesion of the particles to the substrate. The gas temperature was 700 °C, the gas pressure was 4 MPa, powder feed rate was around 20 g/min, gun traverse speed was 300 mm/s and the standoff distance was 80 mm.



316L



CP Fe

Fig. 1. As received powders

The 'coatings' were removed from the substrates by electric machine discharge prior to mechanical testing and annealing. Annealing was performed to increase the ductility of the metals. To minimize oxidation, the samples were wrapped in a steel sheet and annealed at 1100°C for 1 h in an argon gas atmosphere. After one hour had elapsed, the sample remained in the oven for a further 30 min while the oven cooled and the sample was removed and allowed to cool to room temperature.

Cross sections were ground to a typical metallographic finish and the porosity was determined using a Nikon Epiphot 200 microscopy equipped with Clemex Vision software.

Microhardness was measured with a Vickers Microhardness tester (Clark, Clemex^{TM} CMT) at a load of 50 gram

Prior to mechanical testing, the samples were polished in order to eliminate any micro-cracks on the surface. Mechanical properties were determined by micro-shear punch testing, which is a quick and easy method to characterize limited volumes of material. The test is based on a blanking operation using a flat, cylindrical punch of 1.55 mm diameter, which is moved at a constant speed to punch a hole in a flat, thin sample,10 mm by 4 mm by 0.60-0.85 mm thick. Load-displacement curves obtained from micro-shear punch testing can be correlated with conventional tensile tests to obtain yield and ultimate tensile strength of the coatings [9].

3 Results

3.1. Compositions of the composites as-cold sprayed

The chemical compositions of the composites were measured and then used to determine the amount of CP Fe and 316L in the coatings. The results reveal that the CP Fe levels are approximately 5 wt% lower in the as sprayed composite compared to the as-mixed powders, for all mixed powders. This may be due to the lower deposition efficiency of the CP Fe compared to the 316L (approximately 30% vs 70%), but it is interesting that the absolute decrease in the CP Fe level did not change with composition of the composite.

3.2. Effect of annealing on microhardness

Figure 2 shows the change in microhardness of the 316L and CP Fe in all the composites. In the as-sprayed state, the hardnesses of both powders are quite similar and remain largely unaffected by tempering at 400 °C. At 800 °C there is a significant drop in hardness of both powders, indicating recrystallization. As well, after annealing at 800 °C and above, there is a clear separation in the hardness values of the component metals, with CP Fe now clearly softer than the 316L.

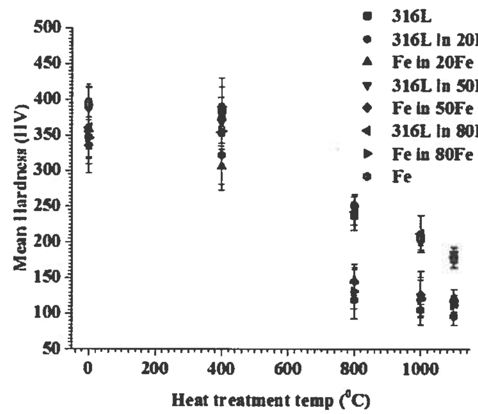


Fig. 2. Effect of annealing temperature on microhardness of 316L and CP Fe in all composites.

3.2. Effect of annealing on porosity

The change in porosity of samples exposed for 1 hour over temperatures of 400, 800, 1000 and 1100^{0}C, was monitored. Significant reductions in porosity start to occur at 800 ^{0}C coinciding with the noticeable hardness reduction and recrystallization. Recrystallization is an indication that diffusion rates are significant and it is this transfer of material that is leading to a reduction in porosity. All composites exhibited porosities less than 0.6% after annealing at 1100^{0}C, but the 50% Fe composite exhibited the lowest porosity, which is possibly related to having the highest CP Fe / 316L interfaces.

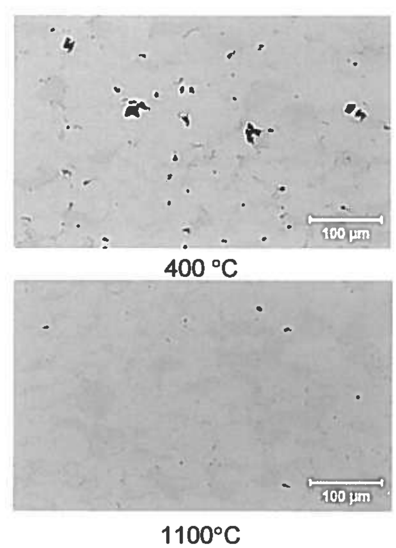


Fig. 3 Effect of annealing temperature for one hour on porosity of 20% CP Fe composite.

3.3. Micro-Shear Punch Test of the Coatings

Figure 4 shows the effect annealing temperature on the flow behaviour of the single component cold sprayed

316L. In the as sprayed condition, the ductility is negligible, and this leads to a very low fracture strength. At 400 °C there is little change but at 800 °C there is a dramatic increase in ductility and a corresponding increase in strength. There are further increases in ductility and strength with increasing annealing temperature.

This increase in properties appears to coincide with the recrystallization observed in the microhardness results. However, recrystallization is a *softening* mechanism. The increase in strength is more likely directly related to the increase in ductility, which, in turn, is likely to be related to the reduction in porosity. However, the level of porosity even in the as-sprayed condition is very low and the observed level of porosity decrese is unlikely to lead to such a dramatic improvement. It is more probable that interparticle 'sintering' has occurred, increasing the ductility in this way. Recrystallization may lead to sintering because considerable movement of atoms takes place, mainly through grain boundary motion. This may help metallurgical bonding to take place at particle to particle interfaces.

Figure 5 shows the corresponding behaviour for single component cold sprayed CP Fe. Once again the as-sprayed properties are very low; 400 °C brings little improvement and 800 °C dramatically improves ductility and fracture strength. In contrast to 316L, however, increase the annealing temperature above 800 °C leads to virtually no further improvement. This suggests that the kinetics of 'sintering' (and/or recrystallization) is faster in CP Fe compared to 316L. Note also that the 316L reaches much higher strength levels than the CP Fe, although the ductilities are similar.

Figure 6 shows the effect of annealing temperature on the shear punch curves of the 80% CP Fe composite. Its behaviour is more similar to the 316L than the CP Fe in that the fracture strength continues to increase with annealing temperature above 400 °C in contrast to the cold sprayed CP Fe. However, there is a fundamental difference in flow behaviour between the composite and either of the single component materials, and this is discussed below.

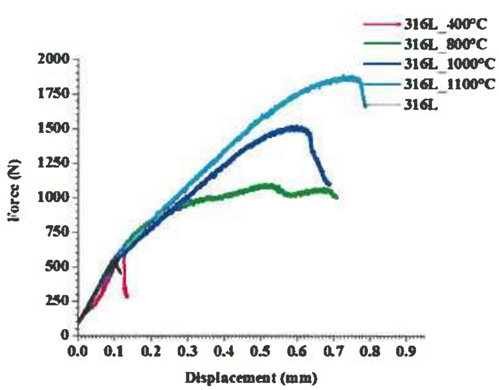


Fig. 4 Effect of annealing on cold sprayed 316L

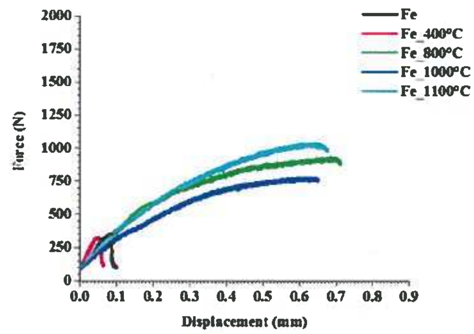


Fig. 5 Effect of annealing on cold sprayed CP Fe.

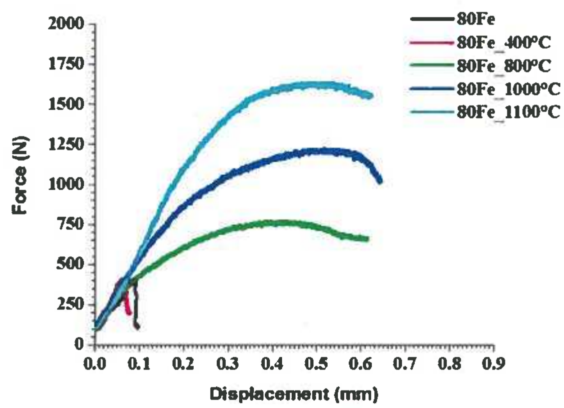


Fig. 6 Effect of annealing on 80% CP Fe composite.

4. Discussion

As noted earlier, the effect of annealing is to increase the strain to fracture, which, in turn increases the fracture strength, primarily through work hardening. This effect is well established at 800 °C for all the cold sprayed materials shown in this paper. For the 316L, increasing the temperature above 800 °C leads to further increases in ductility and strength, but note that all the curves fall on top of each other initially and deviate from each other only when fracture takes place. In other words, the *plastic flow* behaviour is identical for each annealing temperature but the *fracture* characteristics differ. Plastic flow is essentially the breaking and reforming of atomic bonds, I.e. dislocation motion, whereas fracture is breaking of bonds. The fact that the flow curves fall on top of each other at low strains suggest that the plastic flow behaviour is the same for the specimens annealed at 800 °C and above, regardless of the annealing temperature, whereas the fracture behaviour is different.

For the same microstructure, any differences in flow behaviour between the annealing conditions would be due to different amounts of work hardening. Work hardening is removed during annealing by recrystallization. Because the plastic flow behaviour is the same, it suggests that recrystallization is more or less complete at 800 °C. If it is assumed that fracture preferentially occurs at particle/particle interfaces, then the increasing fracture strain with increasing annealing temperature above 800 °C is probably due to increasing levels of 'sintering'. Normally, sintering, in classical powder metal processing, is primarily a means to reduce porosity, but at the same time metallurgical bonds are formed across the particle/particle interfaces. For these as- cold sprayed powders, porosity is low and not a first order issue, but metallurgical bonding between particles is limited. Annealing at high enough temperatures will rapidly sinter the material by increasing the metallurgical bonds.

In the case of CP Fe, annealing at above 800 °C does not significantly change the plastic flow or fracture behaviour, indicating that 'sintering' is easier in CP Fe. This may be due to the absence of the tenacious Cr oxide film on the powder particle surfaces, which is part of the reason why 316L is 'stainless'.

In the case of the composite, like the 316L, the fracture strength increases with annealing temperatures above 800 °C, but there are two differences: (i) at low strains, the curves do not fall on top of each other and (ii) the fracture strain does not increase with annealing temperature. Since it appears that 800 °C is sufficient for full recrystallization of both 316L and CP Fe, the change in fracture strength with annealing temperature is probably mainly due to sintering of the particle-particle interfaces.

There are three types of such interfaces in the composite: CP Fe - CP Fe, 316L - 316L and CP Fe - 316L. Therefore, at 800 °C, it can be assumed that the fracture strain is mainly due to the bonding of CP Fe particles to each other and the flow behaviour is due mainly to CP Fe. At 1000 °C, the mixed metal interfaces bonding is 'complete' and flow behaviour includes some 316L deformation. At 1100 °C, 316L - 316L bonding is 'complete' and all the 316L in the composite contributes to the flow behaviour. Thus the increase in flow strength of the composite is directly due to an increasing amount of 316L contributing to the load bearing of the composite, as opposed to increasing the metallurgical bonding between particles per se. Clearly, the UTS in a metal composite is not directly proportional to the volume fraction of the component metals since the UTS of the 80 % CP Fe composite is almost the same as the 100% 316L specimen after annealing at 1100 °C. However, it is difficult to explain why the fracture strain seems to be independent of the increase in particle-particle sintering. Perhaps the strain to fracture is predominantly controlled by the CP Fe – CP Fe bonding, since CP Fe constitutes 80% of the composite and the CP Fe particles are all sintered at 800 °C.

5. Conclusions

Composites of 316L and CP Fe were successfully made by cold spraying, although all composites exhibited a CP Fe content approximately 5% lower than the composite in the as-mixed powder condition.

The porosity of the as-cold sprayed coatings was low and fell to less than 0.6% for all materials after annealing at 1100 °C. The 50-50 composite exhibited the lowest porosity in the annealed condition.

Annealing at 800 °C significantly lowered porosity, drastically reducing the hardness of both the 316L and CP Fe (whether in the single component material or in a composite) and radically increased fracture strength and strain to fracture.

Much of these improvements are associated with recrystallization, which in turn is due to large scale movement of atoms. But recrystallization per se is not thought to lead to increases in fracture strain and stress. It is suggested that sintering, which is also due to large scale atom movement, leads directly to the increased fracture strain and stress.

There is considerable potential in terms of increasing mechanical properties by mixed metal cold spray strategies since a composite containing 20% 316L almost attained the fracture strength of 100% 316L, which represents a factor of 3 increase compared to the fracture strength of 100% CP Fe.

6 Literature

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