Ordering kinetics evaluation of FeAl powders

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\textbf{A R T I C L E I N F O}

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\textbf{A B S T R A C T}

In this study, time resolved X-ray diffraction experiments using synchrotron X-ray radiation have been performed to get insight on the time and temperature dependent atomic ordering of an intermetallic Fe-40Al (at.\%) ball-milled powder. The target of the present study is to gain knowledge on the rapid heating processes occurring during Thermal Spray coating processes. Present results show that in the temperature range 400 °C - 550 °C, the evolution of the order can be followed and modelled by fitting the powder diffraction patterns collected within the first minutes after fast heating. Reasonable refinements have been obtained by assuming the presence of two domains corresponding to the ordered and disordered lattices. The lattice constant changes from 0.29165 nm in the ball-milled powder at room temperature to 0.29281 nm in the ordered phase after 3000 s at 550 °C. The growth of the ordered phase is proposed to be a vacancy-related process with an activation energy of 1.04 eV. Above 550 °C, the ordering kinetics appears too fast to be resolved using the few seconds time scale of the present experiments which is in agreement to thermal spray results conditions.

1. Introduction

Ordering phenomena is a key process for the proper understanding of the outstanding properties that intermetallic compounds can offer in terms of mechanical, electrical and magnetic properties. This has been investigated for decades in the attempt to elucidate how slight changes on crystal lattice order result in changes on these properties \cite{1-3}.

The order-disorder transitions take place at a so-called critical ordering temperature \( T_{\text{c}} \), which exists for either first or second-order phase transitions. At temperatures above \( T_{\text{c}} \), a short-range order (SRO) parameter is defined and the atoms are prone to order over short distances only; below \( T_{\text{c}} \), a long-range order (LRO) parameter, defined by the tendency of atoms to be surrounded by unlike atoms, shows a dependency on increasing the temperature i.e. it decreases continuously like CuZn or drops abruptly to zero slightly above \( T_{\text{c}} \) like Cu₃Au \cite{3}.

Certain alloys possess the ordering temperature close to the melting temperature \( T_{\text{m}} \). Such alloys keep therefore a permanently ordered structure unless a special treatment such as high deformation by ball milling or high dose particle irradiation is applied \cite{1}. Representatives of this group are Ni₃Al, Ni₅Al, FeAl, Ti₃Al; by contrast, examples of reversibly ordering alloys, whose \( T_{\text{c}} < T_{\text{m}} \), can be found within Cu–Pt and Cu–Au systems \cite{4}. From the first category, transition metal aluminides have been mainly investigated due to their low densities and promising high temperature oxidation resistance properties.

But how then is their degree of order affected upon fast heating up to almost the melting point followed by rapid cooling? This concept is interesting to elucidate what happens to Thermal Spray coating processes. These are a group of technologies where sufficient kinetic and thermal energy are imparted to the raw material (in powder, wire or rod form) to create a confined high-energy particle stream, and propel the energetic particles toward the substrate using a high-pressure carrier gas. The molten or partially molten particles deform on impact with the substrate creating cohesive bonds with each other and adhesive bonds with the substrate \cite{5,6}; even just kinetic energy can be transferred to the particles in the more recent less conventional Cold Gas Spray process, inducing solid-state bonding only by suitable plastic deformation of the material \cite{7}.

Due to the thermal history of the powder upon spraying, which can involve particle temperatures near the melting point of the material (specially in conventional processes) and afterwards high cooling rates upon impact onto the substrate, partial ordering can be obtained in intermetallic coatings \cite{8}. The main motivation of this study is then to evaluate how fast a disordered intermetallic powder becomes ordered due to rapid heating, in analogy to what happens to particles in the flame. We expect to gain insight of time-temperature dependent atomic ordering in the processes involved during the spraying, oriented to improve the final properties. Since usual x-ray data acquisition with...
proper resolution takes a long time, synchrotron radiation will be used.

Nano-crystalline Fe-40Al (at. %) structures are obtained by thermal spray from a ball milled feedstock powder as a way to enhance mechanical properties. We have produced Fe-40Al (at. %) coatings by the high velocity oxy-fuel (HVOF) technology, obtaining good corrosion and oxidation resistance [9,10]. Transition metal aluminides have strong potential for replacing superalloys and stainless steels in moderate and high temperature structural applications. FeAl-based alloys possess an outstanding high-temperature corrosion resistance [11] and they have a lower density (5.56 g/cm³) compared to other iron-based materials such as cast iron and stainless steels (e.g. in heating elements, hot-gas filters, piping and tubing, exhaust manifolds, etc.). However, some aspects such as poor room temperature ductility and toughness and mediocre creep strength, as well as fabrication difficulties have hindered the introduction of intermetallics in many cases as industrial structural materials [12]. Because of all these reasons, aluminides appear particularly interesting as coatings on more conventional higher-strength materials which are less corrosion-resistant at high temperatures.

Many works have dealt with the order-disorder transitions in the FeAl system in terms of structural changes upon temperature, mainly through magnetic, calorimetric and X-ray diffraction experiments, showing complementary results with these techniques [13,14]. Some authors have pointed out that the strength of ordering behavior of late transition-metal aluminides finds its origin in the atomic size effect, since the reduction in strain energy due to atomic size misfit can be achieved by ordering. However, the validity of this size effect has also been questioned for years [15].

Owing to long data acquisition, conventional laboratory X-ray sources present serious limitations for the study of how the order parameter in thermal equilibrium varies with temperature. On the other hand, the high intensity offered by synchrotron X-ray radiation sources coupled with modern fast timing detectors, allows getting insight on the kinetics of time-temperature atomic ordering processes. Such techniques are being applied for investigating the ordering transitions [16–19] as well as the evaluation of several material crystallization kinetics [20–23] and other reactions [24–27]. In their work on Cu3Au, Shannon et al. studied the order-disorder transition starting from a disordered state at an initial temperature Tt above the critical one (Tt > Tc) and then qenching into a region of the phase diagram where the equilibrium state is ordered (Tt > Tc) Tt standing for final temperature [19]. By contrast, Schaefer et al. performed some time dependent experiments for Fe-45Al (at. %) specimens by using time-differential length scale measurements after fast heating rates and then left isothermally treated; they obtained the vacancy formation and migration enthalpies, 1.0 ± 0.1 eV and 1.5 ± 0.2 eV respectively [28]. The purpose of the present study is to report the ordering process in a Fe-40Al (at. %) ball milled powder alloy upon fast heating from the room temperature disordered state and, to evaluate the kinetics of such ordering process as well. As mentioned above, this will help with the understanding of the ordered phase formation in thermal spraying.

2. Experimental method

A ball milled Fe-40Al (at. %) alloy powder supplied by CEA-DTEN (Grenoble) was used for this study. It has a nominal composition of Fe-40Al-0.05Zr (at. %) + 50 ppm B+1 wt%.Y2O3. Also an atomized powder was used for comparison.

The temperature dependent and time resolved powder diffraction experiments were performed on the BL04-MSPD beamline of the synchrotron facility ALBA located in the Barcelona area [29,30].

The powders were enclosed into sealed quartz capillaries of 0.5 mm diameter, and the data was collected in transmission geometry using a monochromated beam of 0.042492 nm wavelength. The set-up included a remote controlled heated air blower (Cyberstar) allowing collecting data at the selected sample temperatures 400, 450, 500, 550, 600, 650, 700 and 750 °C after fast heating from room temperature. The hot spot of the blower, this is the constant temperature range of the sample, is 5 mm in length and the X-ray beam was 3 × 0.7 mm². The time needed for the blower to go from room temperature to the selected data acquisition temperature was about 3–5 min, also considering a non-negligible temperature overshoot of ~5%. The analogy to thermal spray appears as an approximation to the process since the powder is heated when fed in the flame and high temperatures are reached in very short times. To avoid the higher temperatures due to the overshoot, the following methodology was applied. The sample was rapidly heated at the blower set temperature and left stabilized during ~10 min. It was then remotely moved 10 mm (10 s moving time) from that position to heat another portion of the sample. By operating in this way, the part of the sample studied was quickly heated at the selected temperature without suffering from temperature overshoot. It was checked that, at that zone 10 mm apart from the initial heated position, the atomic order did not change, even testing at the highest temperature. The time for the 0.5 mm diameter capillary to achieve the final temperature, when laterally moved and exposed to the hot air flow, was estimated to be around 10 s. Sufficiently good data statistics could be obtained with 5 s effective acquisition time, which taking into account data processing/transfer and temperature reading resulted in 8.2 s time resolution of our X-ray powder data collection. The powder patterns were registered using the position sensitive MYTHEN detector (6 single modules of 1280 channels) allowing collecting in a single shot a 41.1° 2θ angular range in 0.0062° steps. Between two detector modules, the pattern is obscured over a ~0.2° angular range; it was hence crucial for time resolved experiments to smartly position the detector so to avoid Bragg reflections on this specific “dead” regions. The X-ray diffractograms will be presented in reciprocal space units instead of the 2θ scattering angle:

$$Q = \frac{4\pi\sin(\theta)}{\lambda}$$  \hspace{1cm} (1)

The X-ray patterns were refined by the Rietveld method using the FullProf software [31]. For this work, we assumed the presence of different domains corresponding to a completely ordered bcc B2 superlattice and a completely bcc disordered phase [32,33]. Since ordering parameters are related to the microstructure (strain and size), a particular attention was focused on the analysis and fitting of the x-ray peak shapes; for that purpose, we used the Thomson-Cox-Hastings pseudo-Voigt function [32]. The instrumental resolution was determined by collecting data of a Na2Ca3Al5F14 sample enclosed in 0.5 mm diameter capillary.

In order to achieve a good correlation between the calculated and the experimental diffractograms, the minimum values of the usual fit indicators of the FullProf software were monitored. The increase of peak full width at half maximum (FWHM) respect to the instrumental
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