

# **Development of an austenitic/martensitic gradient steel connection by additive manufacturing**

**Dr. Flore Villaret, CEA/EDF, France (Winner of the 2021 Pitch Your Gen IV Research Competition)**

## **Berta Oates**

Welcome, everyone, to the next Gen IV International Forum webinar presentation. Today's presentation is on the Development of an Austenitic/Martensitic Gradient Steel Connection by Additive Manufacturing, represented by Dr. Flore Villaret.

Doing today's introduction is Dr. Patricia Paviet. Dr. Paviet is the group leader of the Radiological Materials Group at Pacific Northwest National Laboratory. She is also the Chair of the Gen IV International Forum Education and Training Working Group. Patricia?

## **Patricia Paviet**

Thank you so much, Berta, for the introduction, and I am very pleased to welcome Dr. Flore Villaret. She recently completed her Ph.D. at the French Atomic Energy Commission in the field of material science. She is now a research engineer at the R&D Department of Électricité de France. She works on developing additive manufacturing of metal components for energy applications such as nuclear reactors and hydraulic power generation. She is also Vice President of the French Metallurgy and Materials Society Young division.

We launched in January 2021, an initiative with this GIF Education and Training Working Group which was the "Pitch Your Gen IV Research" competition, and she won this prize with a very creative and original video presenting her Ph.D. work in additive manufacturing metallurgy for Gen IV reactor. She was also awarded by the French metallurgy and material society with a Bodycote Best Ph.D. Thesis award. Thank you again, Flore, and congratulations on winning this prize. I give you the floor, and I cannot wait to listen to your presentation. Thank you.

## **Flore Villaret**

Thank you, Dr. Paviet.

Before beginning this presentation, I would like to thank all the people who helped me to realize the work that I am going to present to you, so thank you.

I would like to start this presentation by talking about the different materials that can be found in a nuclear reactor. This is a Gen II or Gen III reactor, but you can have the same materials or the same ID with Gen IV reactors. In a nuclear reactor, the parts are subjected to stresses that can be very different depending on their location. For example, there are large differences in temperature between the water of the primary circuit which is in this case is around 300 Celsius degrees and the ferrite setting which is around 650 Celsius degrees.

The materials in the reactor will also be subject to corrosion and depending on the location in relation to the field to irradiation. Different materials are required depending on the allocation in the circuit. For example, you can have some martensitic or bainitic steel. The vessel is a good example of components made of bainitic steel. There are also some austenitic steel with the pipe of the primary circuit or the core structures.

You can have also a nickel base alloy with the pipe of the steam generators and also other materials like the zirconium alloy of the cladding tubes. All these different materials need to be joined together and for nuclear reactors, it's mainly done by welding. As you can see, there are also many different metallic materials, so there is a possibility to use metallic additive manufacturing.

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Indeed, some parts have already been produced by additive manufacturing in order to demonstrate the relevance of this process for nuclear applications. Today, at PBF, we already built and put some parts in nuclear reactors. For example, you have this valve stopper or this manual control, which was an obsolete part. We also made some tools for fuel handling.

We have some project like the European nuclear bomb project, which aims to study the behavior of this part in nuclear environments. These additive manufacturing processes are being studied for the construction of parts in the reactors, also for the future reactors like Gen IV reactors. Here, you have some examples of parts, which have been made at the CEA for Gen IV reactors. You have here sodium fast reactor, sodium flow grid. Here, you have also an SFR fuel storage device. Here you have a pipe connection added on a tube.

I don't show an example here, but I know that Oak Ridge Laboratory in the US also made some amazing parts by additive manufacturing to study the possibility to make some parts for nuclear applications.

The study that I conducted during my Ph.D. is quite generic and can be applied to present and future nuclear reactors. I choose to use an example of components located in a sodium fast reactor. It's a component located in the core of the sodium fast reactor in order to have a comparison point with a real part, so to have some properties to compare.

The components that I studied is the joint between the hexagonal tube and the spike. In the sodium fast reactor core, the fuel rods are grouped and maintained in the core by the hexagonal tube made of 9 chromium steel, which is martensitic steel, in order to have good mechanical strength at high temperature and resistance to swelling under irradiation. This hexagonal tube is joined to the assembly spike, which is made of 316L austenitic steel, and this spike is interlocked with the austenitic steel base. In my Ph.D., I studied this junction between the martensitic steel and the austenitic steel.

If you perform this junction by welding, without any filling metal, you can use the Schaeffler diagram to try to anticipate the risk, the cracking risk in the weld. On this diagram, you can see that you have the chemical composition on these two axes, and you have some areas, growth areas which correspond to the different cracking mechanism. You have the 316L austenitic steel here which is in the white area, so no cracking problem and the 9-chromium steel, which is in the purple area, which means we have martensitic cracking risk.

If we join these two alloys together, we have the welding area somewhere on the dashed line depending on the dilution. You see that we have a risk of cold cracking in the melted area. We will need to perform a pre-heat treatment to pre-heat the parts before the welding, and we will also need to perform a post-welding heat treatment for martensite tempering.

The solution, which was used to make this assembly as industrial solution, is to perform Tungsten Inert Gas welding with a filling metal, which is inconel 82. If we come back to the Schaeffler diagram, our inconel 82 is somewhere here above the diagram. Using this filling metal in our welded area, we'll shift the composition of the welded area to rather this arrow, somewhere

here, so we don't have any more the cold cracking risk, but we are in an area with hot cracking risk. This means that we will need to have a really good process control in order to avoid the risk of the cold cracking risk.

This solution was used especially for the assembly of the French SFR Phenix. In this reactor, they used a joining part made of 316L which was welded on the 9-chromium part with filling metal and then the 316 joining path is joined to the assembly spike when you are in the reactor.

My work was about studying alternative solution to this TIG welding. During my Ph.D., I studied a different alternative solution. The first solution was the electron beam welding technique. I studied the dissimilar electron beam welds. In this case, we have the two parts, which are directly weld with hot filling metal by electron beam, and I also studied alternative solution on one hand with traditional powder metallurgy. We made graded part by Spark Plasma Sintering or Hot Isostatic Pressing.

For each of these processes, I studied some homogeneous materials, direct assembly, some mix, and some mix assembly. The idea is to make, for example, here you can imagine to make graded material and in order to perform similar welds on each side of the assembly.

The second alternative solution with powder metallurgy is additive manufacturing. In this case, the idea is more or less the same. The idea is to make a graded connection with additive manufacturing or with this connection, this graded part directly on one hand by the Direct Energy Deposition process or the Powder Bed Fusion process.

For each of these processes, I studied the homogeneous materials and different type of graded parts, more direct or more progressive. As you can see on the screen, the idea is to, for example, build the graded material directly on the hexagonal tube and then perform only one similar weld to the assembly spike.

Today, I will not have time to present all this work, but I choose to present to you the additive manufacturing part. Here is the outline of my presentation. First, I will talk about the materials, the base material, the powder that I used for the study. Indeed, with the additive manufacturing processes, the powder is really the beginning material. We need to well understand the powder before performing some builds [ph].

Then, I will talk about the additive manufacturing process. I will explain you which are the differences between the two processes that I studied and how it is working, and I will show you some results about the 316L and the 9-chromium steel alloy, so only homogeneous materials. Then, I will show you some results about graded materials. In the end, I will conclude and give you some perspectives of this work.

First, let's start with the powders used for this study.

First, here I have put some results of the characterization of the 316L powder. Here you have the chemical composition of the powder, so it's an iron-based alloy. You have some chromium between 16 and 19% for the stainless properties. You have some nickel between 10 and 13%. This is in order to obtain the austenitic structure, so it's a cubic-centered crystallographic structure and you have the L of the 316L which means low carbon, so you have a maximum of 0.03% of carbon.

If we look at the powder with the scanning electron microscope, we can see that this powder is very spheric and this is important because it can show a good probability of the powder, and this is important in order to spread the powder correctly.

We also performed some tomography on this powder. Tomography is like X-ray images of your powder, and this image shows you that our powder is full, with no holes in it. We also measured the repetition of the sizes of the powder here. You can see that the average diameter is around 18 micrometers. It's a good size for additive manufacturing.

Here, you have some EBSD maps of the powder grains. With the EBSD, you can image the crystallographic orientation of the crystals in the powder grains. Here, you can see that we have many different crystals with many different orientations.

Now, if we talk about the 9-chromium steel, you can see the chemical composition here. It's quite different from the 316L. We have only around 9% of chromium and 1% of molybdenum. We have around 0.1% of carbon. We have much more carbon than in 316L, and we have, in this case, a martensitic structure, so it's cubic-centered crystallographic structure, and this is a good structure for the resistance to irradiation swelling and to obtain the satisfying mechanical properties under the service temperature.

The other point is that this alloy has different phases, depending on the temperature, so at room temperature, it is ferritic or martensitic.

But if you increase the temperature around 800 degrees, you start to have the austenite structure. If you increase again, here you have also the data of ferrite structure. That's very different from the 316L which is always austenitic, and you will see that we'll have some consequences on the result observed after.

If we look more closely at the powder, we can see here we again have some very typical powder. On the tomography here, you can see that we have some holes in the powder. That could be a problem because sometimes these holes are full of gas, and you can sometimes observe the gas. Sometimes the gas stays trapped into your box. You still have some porosity in your box because of the gas trapped inside the powder.

Here, you have the repetition of the sizes of the powder. You can see that this powder is smaller than the 316L powder with a mean diameter around 30. If we look at the EBSD map, so the crystallographic orientation inside the powder grains, you can see that the big powder grains are martensitic with many different crystallographic orientations and the small powder grains are only few grains, few different crystallographic orientations and they are mainly ferritic. I will not go further on this point now, but I will explain you why we have these differences later. I will use it to explain some things about the additive manufacturing microstructures.

Now, we know well our powder, we can go through the additive manufacturing process. In this work, I compared two different processes, the Powder Bed Fusion process, and the Direct Energy Deposition process. In each case, we have a laser beam, which is used to melt the powder, but the way the powder is brought is different.

In the Powder Bed Fusion process, you have a building plate where it is spread the powder bed. The first step is to raise up the powder supply, so the recoater can spread a layer of powder on the building plate. Then, you have the laser beam, which will melt the powder at the place where you want to build your part. Then, the building plates can go down and the cycle is starting again. You can see on the video, the recoater and the laser which is melting the powder. Your part is doing it layer by layer.

With the Direct Energy Deposition process, the powder is in a powder feeder and the powder is carried by a vector gas to the nozzle. Then, the powder is projected with the gas under the laser beam and the laser beam melts the powder and welds the part layer by layer. If you look on the video, you can see the powder grains,

which are melted here. You can see that in the end you have some similarities in this process and some differences. The main differences are also the energy density of these two processes. With the Powder Bed Fusion process, you have volume energy density, which is really higher than with the Direct Energy Deposition process because you have a smaller laser head and a smaller laser spot.

Now, if we look at the microstructure, you can obtain with these two processes. I have put here the 316L microstructure. In each of these processes, you have austenite as in the forge parts, but you can see that the microstructures are very different from processes to another and are also very different from a forge part.

With the Powder Bed Fusion process, you have very big grains oriented along the building direction with also crystallographic preferential orientation, so the 0.11 [ph] orientation. With the Direct Energy Deposition process, your grains are smaller. You have some grains oriented along the building direction but also some grains which are more relaxed [ph]. Even if these microstructures are very different one from another and from the typical forged microstructure, these microstructures are quite typical for additive manufacturing.

If we look now at the 9-chromium steel, we have some strange microstructure. By the Direct Energy Deposition process, we have the martensitic structure with all the small grains that we can expect with this 9-chromium steel. This is quite comparable to the forged microstructure but with the Powder Bed Fusion process, it's more surprising. We have some big grains and small grains. In fact, it's because we have a mix of ferrite and martensite inside your materials instead of the fully martensitic structure.

We were quite surprised of this result because usually the ferrite is obtained only with a slow cooling process and additive manufacturing is all the opposite of that, it's really a very fast cooling process. We said, okay, we need to perform an in-depth study to really understand this microstructure.

Now, I will present to you the result of the study and try to explain you why we have such difference in the microstructure. Before beginning a big experimental study, I started to look at the literature for other example or experiment about this steel. I found this paper, which is on additive manufacturing of reduced activation martensitic steel, so it's 9-chromium, 1-tungsten. It's very close from my 9-chromium, 1-molybdenum material and by DED and we

see that the authors obtained a martensitic structure as my structure.

After I looked at other publications and I found these two publications. They are on the 17-4PH steel. It's a bit different from my 9-chromium steel but we are still on a martensitic steel which is normally martensitic easily on the forged part. If we look at the PBF samples, in this case, we have the same processing parameters on this picture and this one, but different powders and we can see that in the case we have the martensitic structure and, in another case, we have a fully ferritic structure. We have a very different structure just depending on the chemical composition of the powder.

Here, in this case, we have the same powder, the same parameters but different wall thicknesses. Again, we have on one hand, a mainly martensitic microstructure and on the other hand, mainly ferritic structure. This is, again, very interesting. This showed me that I was not alone to obtain such strange results.

Now, I will try to explain you why we have these differences and these phenomena.

First, in order to understand what happens, we go back to something simpler, the powder microstructure. If you remember, I said that in the small powder grains, I have ferrites and in the big powder grains, I have martensites, and if you look at the intermediate size of the grains, I have a mix of ferrite and martensite.

In the literature, there are not so many papers, which are studying the powder grains microstructure, especially on martensitic steel but I found this paper. It's again on the martensitic steel powder and they observed quite the same thing. They observed some ferritic microstructure in the small group of the grains and some martensite in the big powder grains.

In these papers, I also compute the cooling speed of the powder grains when they are formed, and as we can expect, the small powder grains cool very fast and cools faster than the big powder grains. That means in the case of the powder, the faster we cool, the more ferrites you get. That's very unusual for the metallurgist as I am. Usually, in metallurgy of martensitic steel, it's the opposite. The slower you cool, the more ferrites you get, so it's quite disturbing.

If we try to make a link between the powder and the additive manufacturing, we can use, for example, this diagram, which is



extracted from a publication where they measure some microstructural parameters to deduce the cooling rates depending on the energy used to build the parts.

If I put the point of my process, you have here the DED with 1 to the 4 Celsius degrees per second and the PBF is here with 1 to the 6 degrees per second. The PBF cools 100 times faster than the DED process for the parameters that I choose. If you remember, in the DED process, I had the martensitic structure and in the PBF process, I have the ferritic and martensitic structure. Again, increasing the cooling rate is decreasing the martensite fraction in additive manufacturing and in the powder but that's valid only for very, very fast cooling rates.

In order to try to understand what happened, we can have a look on the equilibrium phase diagram. If you assume the thermodynamic equilibrium and start from a liquid, we first have the formation of delta ferrite, so the liquid solidifying delta ferrite. Then, the delta ferrite transformed into austenite. Then, the austenite transformed in ferrites which is usually replaced by martensites. But with the additive manufacturing, you see that we have very fast cooling rates, so we are out of equilibrium and the PBF structure is a very good example of this out equilibrium behavior.

If we try to understand what happened, the question is, finally, can we bypass the austenite during the cooling of the materials? If we solidify in delta ferrites, can we just never transform in austenite or is it possible or not? To answer this question, I will show you that, finally, it is mainly the time spent between the Ae5 temperature and Ae1 temperature which controls the austenite formation.

First, let's assume that we have an austenite nucleus, which is immediately formed at Ae5 and let's study how it will grow. The austenite growth is mainly controlled by diffusion. Here, we have very fast growing grain. Only interstitial elements, like carbon or nitrogen will have time to diffuse. In my 9-chromium steel, I have only 4 PPM of nitrogen and 1000 PPM of carbon. We will only consider the carbon diffusion.

I can compute the ranges of the size of a new austenite grain at Ae5 during the cooling. Here, you have the diffusion coefficient of the carbon and nitrogen, the temperature which depends on the temperature, and here you have the cooling rates. The time that you will pass in the Ae4-Ae3 domain.

With this equation, we can plot the size of the new austenite grain when Ae3 temperature is reached depending on the cooling rate or

the time span between Ae5 and Ae3, and you can see that there is a strong effect of the cooling rates on the growth of this austenite nucleus.

If we compare with the DED on the PBF cooling rates, you can see that in DED the time spent between Ae5 and Ae3, so it's like 60 milliseconds, is sufficient to allow the austenite to grow until the delta ferrites disappear. You can have big austenite grain but in PBF, you have very faster, really faster cooling rate, so the time spent between Ae5 and Ae3 is really too short. You have only 6 milliseconds, and you have only small grains that can be formed, and the delta ferrite remains.

If we try to sum up all this information and make a link with the microstructures that I observed, we can start with this diagram. It's the CCT diagram of the 9-chromium steel, the usual CCT diagram of the 9-chromium steel. Usually, if you cool slowly, you have the austenite, then the austenite transformed to ferrites, and if the austenite remains in martensite, then you have mainly alpha ferrite.

If you cool fast, your austenite is not transforming to ferrite and transformed immediately into martensite when the MS temperature is reached. But as you see, in additive manufacturing, we have very fast cooling rates, and we need to consider that we are not starting from the austenitic domain but from the delta ferrite domain.

In this case, in additive manufacturing, for example with the DED process, so with not so fast cooling rates, you start from the delta ferrites, transform into austenite, and then from the martensite. But with the PBF process, the cooling rates are so high that the delta ferrite never transforms into austenite, so you keep the delta ferrites until the end of the cooling. If we modify the chemical composition of the powder, you can increase or decrease the gamma domain. In this case, it is decreased for example and change the microstructure. In this case, the DED is not anymore crossing the gamma domain, so we have the delta ferrites always.

This shows you that a very precise control of the chemical composition and the building parameters is required. Chemical composition, as I showed you, can change the size of the domain and building parameters churns the cooling rate. This control is required to obtain the good as built microstructure in martensitic steels. This model could be used to set a relation between the composition and cooling speed to control the microstructure.

All these questions are important because delta ferrite is usually avoided in welds mainly because it decreased impact strength and lowered mechanical properties after aging. The other point is that we have a better resistance under irradiation of martensitic structures. All the results that I showed you about this question of cooling rates and martensitic steel was published in the material here. You have the reference here if you want to have more details about these two.

If we go back to the PBF microstructure, we can try to understand the microstructure with the things that I explained previously. If we look at the last layer here, we have a ferritic layer. This last layer undergoes only one thermal cycle. The powder is melted, then solidified in delta ferrite, and it will transform into austenite. We have only delta ferrite.

If we look at the layer under, this layer also was melted and solidified once but this layer was also reheated when the last layer was built. We have a new austenitic transformation here and here. We have more time to transform austenite and have some martensite after, and if we look at the layer here, these layers have undergone several thermal cycles as they were austenitized several times, so we have more martensite here.

Now, as we understand better the formation of the microstructure in the homogeneous materials, we can go to graded materials. Before showing you the result of the graded materials, I would like to talk about the link between the composition and the microstructure. You can try to anticipate the microstructure with the Schaeffler diagram.

Here, you have the 316L which is in the austenitic area, so austenite with a small amount of ferrite and the 9-chromium steel which is in the martensitic area or martensite plus ferrite, a small amount of ferrite, and so if we have a graded area between these two materials, we will have a change from austenite to martensite and many different microstructures in few millimeters. Here, we can also have austenite, martensite, and ferrite mix.

We can also use thermodynamic calculation to anticipate the temperature of phase transformation. I compute the pseudo-equilibrium diagram of the 316L 9 chromium steel. You have the 9-chromium steel here, the 316L here, and you can see that the transformation temperatures are changing depending on the chemical composition.

First, let's try to make some gradient material by Powder Bed Fusion. It's not so easy because our Powder Bed Fusion setup was not really made to build some graded parts because we have only one place to put the powder. To make some graded materials, we need to put one powder upon another and spread the powder directly together. We fill the powder reserve with two materials and build with this technique.

With this technique, it was possible to obtain a junction without the defects, and it is possible to control somehow the extent of the gradients by controlling the filling methods. For example, if you make some spike like this, your graded area will be wider than if you make just a flat change. If we look at the microstructure that we can obtain with such technique, you'll see that we change from the austenitic microstructure on the 316L to martensites in the graded area and to the martensite plus ferrite microstructure that we observed previously.

Now, if we use the DED process, it's really the good process if you want to make graded materials because you can have several powder feeders. In my case, I used two different powder feeders, one with the 9-chromium steel and one with the 316L steel. You can control the composition at each layer with the powder flow. The amount of powder you send in the nozzle, you can really control the chemistry at each layer.

In the case of my study, we optimized the building parameter for the 316L alloy and as the 9 chromium is, it's a different steel but we are still with iron-based alloy, so we kept the same parameters for the 9-chromium part. The power and the speed were kept constant for the two alloys, and only the powder flow and the dilution varies from a sample to another to control the layer height. As I said, the composition is controlled with the powder flow.

With this technique, we also succeeded to obtain some materials like this one. This is a sample with the 316L down and the 9 chromium up. They don't have the same powder flows without any cracks or any defects.

Before showing you the microstructure results, I would like to talk about little the dilution in additive manufacturing. In additive manufacturing, the dilution can be defined as the overlap rates between two beads. It's like a re-melting rate. This concept is a lot used in welding and especially dissimilar weld. If we look at the scale here, let's imagine we are building layer "n" with the 316L. If we build on this layer 9-chromium steel layer, we will re-melt a part

of the 316L layer and this re-melted area will mix with the powder that we used previously.

The composition of the layer we are depositing is not 9-chromium layer. It's a mix between 9 chromium and 316L and this mix will depend on the area which is re-melting. In this case, if we have low re-melting or high re-melting, we have different results. This dilution can be computed with this formula so we can use the layer height and the difference of "h" between two layers. Then, with this dilution, we can compute the composition of the n plus 1 layer with this formula.

In my study, we kept all the parameters constant. We kept the power and the speed constant, and we have only the powder flow which is varying and so the dilution which is varying from 80% to 50%.

With this equation, we can try to anticipate the chemical composition of the gradients. If we assume a dilution of 80% and a direct change of powder, so we first build with the 316L powder and then build with the 9-chromium powder, we can have a very gradual change, even if we change directly the composition because of this 80% dilution, so it's the yellow curve.

If we perform the same build but with 80% of dilution, the change is more abrupt and more fast. In this case, it could be useful to introduce a layer of intermediate composition. That's the idea of the blue curve. On the blue curve, you have 50% of dilution but you have a gradual change in five layers, so during five layers, I will mix the two powders together in order to try to have a more gradual change. You'll see that we can obtain a really more gradual chemical change.

I will not present all the results of these different cases, but I will just talk about the more granular gradients that I obtained. These are the gradients with 50% of dilution and a gradual change in five layers.

Here are the microstructure and the chemical profiles that we can obtain in these samples. Here, you have, so you can see that we have the gradient over several millimeters. Here, the distance is in micrometers, so you have 5 millimeters of graded area. This is the chemical composition in chromium along the gradients. You have in light yellow, the calculated profile and in dark yellow, the real measured profile with the EDX process and you can see that there is a good correlation between the two profiles.

The methods are that, as we show you, to anticipate the gradient is really useful and if we look at the microstructure, you can see that we have the change from the austenite here in green or here to the martensite here and here progressively. We have really the evolution of the austenite fraction, which is along with the chemical composition.

After if we perform some micro-hardness in order to try to evaluate the local mechanical properties, we can see that we have very big differences of hardness in our graded area. On one hand, we have the quite soft 316L austenitic structure. On the other hand, we have a harder martensite but martensite with 300 Vickers is a quite soft martensite in fact. Between these two pure materials, we have the graded area. We can see that it becomes harder here and then, we have a maximum of hardness here before getting a softer material.

This change of hardness is directly correlated to the martensite fraction. As you can see here, the more martensite you get, the harder you will be and also to the chemistry and the phase transformations that can occur in the reddened area which are not occurring on the 9-chromium part. You have really this area of the gradient which is harder than the rest of the material.

Now, if we try to sum up a part of my work and compare the different gradients. I tried to put on only one graph all the chemical profile of all the gradients that I studied. In black and grey, you have the DED gradient, so a change with five layers, with a very long gradient, and a direct change of high dilution which are smaller. In red, you have the electron beam weld profile, and in light green, you have the HIP profile, in dark green you have the Spark Plasma Sintering profile and in yellow, the PBF. We have zoomed here in order to compare these three profiles.

You can see that with the DED, we can really have a wide control of the chemistry in the gradients through the parameters and the dilution, so the parameters and dilution and the introduction of powder mix. It is possible to obtain very short gradient with PBF because we have low layer height. With the Spark Plasma Sintering and Hot Isostatic Pressing sintering processes, we can also have a very short gradient because in this case, the gradient is only controlled by the diffusion. We have a possibility to control and anticipate the chemistry and the length of the gradients in DED either by the blown composition, so the chemistry that we are using, and the dilution rate, so the manufacturing parameters.

Now, we are reaching the end of the presentation. I will just make a small conclusion and give you some perspective of the works. First, about the microstructure. Here, I have put all the different microstructures that I obtained during my Ph.D. work. Here, you have the EB weld, Spark Plasma Sintering, DED gradients with no transition and low dilution and PBF gradient. We saw that we have really a relationship between composition and cooling rates.

As I explained, we can really have a microstructure control in martensitic steel with the composition and the cooling rates. On all these gradients, we see that the metallurgical continuity is assured between the two materials. We have no cracks or no defects. We have very diverse microstructure depending on the process use, so only local melting by electron beam welding.

We can have transformation of the whole material without melting with the Spark Plasma Sintering or Hot Isostatic Pressing or we can have transformation of the whole material with melting in additive manufacturing. I showed you quickly also that we have really good links between chemistry microstructure and microhardness, and I showed you also that we can predict and control the chemistry in the gradient with the DED process.

I also performed some tensile tests in order to evaluate the mechanical property of this junction. Again, we have in the red the EB weld, black and grey, DED gradient and the light green, HIP and dark green, Spark Plasma Sintering. I performed the tensile test at room temperature and 550 degrees. In each case, we have all the similar macroscopic behavior between the EB welding and the other kind of gradients.

At room temperature and at 400 Celsius degrees, the failure which happens on the 316L side because this material is softer than the 9-chromium steel at this temperature and at 550 degrees, the failure occurs on the 9-chromium side because at higher temperature the 9-chromium steel becomes more ductile and is less softer on the tracing [ph]. In each case, it is important to note that the failure occurs in the base material and not in the graded area or in the weld. That's very important because it's like the key points for evaluating this junction.

These results are very encouraging, if we imagine, to use this material in an industrial context, but obviously, we need also more complete evaluation of this junction.

Here are some proposals for future study. Some examples of proposals that we can further study. We can think about evaluating

the toughness of the graded area. Indeed, as we saw, the gradient area is harder, so we can wonder if we have a brighter area or not. The toughness could be very useful. We can also think about carbon diffusion and aging behavior, maybe introduce a barrier material.

There was already one study which was performed. It's this study on material with bimetallic junction with weld with inconel 82 or graded area and they showed that the graded material really helped to slow down the carbon diffusion and increase the aging resistance of the junction.

We can also think to perform corrosion. Again, there was some study on materials, which are closed for my materials, here for example, and they showed that the change of corrosion behavior is very progressive but, in this study, the martensitic steel was also stainless steel. Our results could be different as our martensitic steel is only 9% of chromium and, obviously, we also did some irradiation tests to revise that the behavior of the gradient area is good and there is irradiation.

This work and all this complimentary study could open up the way to the fourth dimension. We can imagine toward the three dimensions, which are those of space or X, Y, and Z to which we add the fourth dimension, which could be that of the chemistry. The idea is that we could have some optimized part not only in terms of shape but also in terms of the right composition at the right place and to allow different microstructure and properties depending on what is required for the parts.

I would like to thank you for your attention, and I am ready to answer the questions if there are some. Thank you.

### **Berta Oates**

Thank you, Flore. Congratulations, again, on the award to present today for your Pitch Your Thesis competition. I'd like to remind everyone how bright of a future we have. You can see the kind of talent and expertise that the next generation is bringing to our industry and it's so inspiring.

Well, questions are coming in. Let's take a look at the upcoming webinars in January, ESFR SMART, a European Sodium Fast Reactor concept, including the European feedback experience and the new safety commitments following the Fukushima accident.

In February, AI, in support of the NE sector, and in March, Scale Effects and Hydraulics: Application to the French SFR. If you have



questions, you can go ahead and type those in. The question came now, and we will take the next few minutes. What do we have in here? There are a few things. I have shared the rights with you, Flore, so you should be able to see them as well in the questions pane.

The first one, hi Flore, thank you for a very good presentation. Very nice pictures. A question on the microstructure comparison for 316L comparing DED and LPB. Do you think the microstructure are a function of the specific manufacturing parameters chosen and not necessarily due to the process itself? That one's kind of long, it'd be easier to see it.

**Flore Villaret**

You are asking if the special microstructure I linked to the process or the parameters that I used? That's right?

**Berta Oates**

Right. The microstructures as a function of the specific manufacturing parameters chosen and not necessarily due to the process itself.

**Flore Villaret**

In fact, it's exactly these points that I want to mean. Yes, with the 9-chromium steel, we saw a very different microstructure on one end with the DED and on the other end, with the PBF. But I think it's possible to obtain the same change of microstructure only with the PBF process, for example. I think it's really linked to the energy density which we are using. This energy density induced a different cooling rate which induced again the microstructure. I think if we use the same kind of energy density as we obtained in DED but with the PBF machine, we will obtain the same martensitic structure.

**Berta Oates**

Thank you. The next one is a note to Flore, but I feel compelled to share it with everyone. I hope that's okay. Appreciating your powder study on the iron 9 chromium 1 molybdenum. Thank you for this detailed work. What other mechanical property factors, toughness, and fatigue studies have been conducted?

**Flore Villaret**

I think there are many things that can be done. We can also think about performing some institute studies like traction under microscope or something like this to well understand the behavior of each different parts of the gradient because as we see with the microhardness, we obviously have different mechanical properties at each place of the gradient. With the tensile or the Charpy

toughness test, we will have only a macro view, but we will not have the understanding of the behavior of each part of the gradient. I think we can also perform such tests.

**Berta Oates**

Thank you. Is EDF using these new materials now for advanced – I am sorry, we are going to have to – is EDF using these new materials now for advanced RX AM parts?

**Flore Villaret**

Gradient materials are not yet used by EDF. For the moment, it's only a research project but maybe in the future we will use it. Today, at EDF, we are just beginning to work on additive manufacturing and the possibility to use additive manufacturing to build some fast nuclear reactors. As I showed at the beginning, we have already built parts with only one material and I think if I remember well, it's only 316L because it's the most known and most studied additive manufacturing material. We are just beginning but maybe in the future we will use a gradient material. Yes. Thank you.

**Berta Oates**

Thank you very much for delivering a very nice presentation. Congratulations on the results. One short question. What about the retained austenite? Did you detect such kind of austenite after the cooling?

**Flore Villaret**

In the martensitic steel, no, but I only performed EBSD. If there are some retained austenite with very small domains, I can't see it. But only with the EBSD, I didn't see some austenite, but I think the 9-chromium steel is not really a steel with retained austenite usually.

**Berta Oates**

Thank you. How are the optimum parameters determined prior to the investigation such as part [ph] size, scanning speed, and laser power?

**Flore Villaret**

Part size, usually, it's fixed parameters because it depends on the setup of your machine, so you don't really modify it. Scanning speed and laser power, I don't really make the optimization because I used the work of other Ph.D. and other studies. But generally, the idea is you first make some single pass test. You make some lines with different parameters to see how your powder and your melt pool behave depending on the parameters. When you have a set of

parameters you are satisfied with, you start to make some surface, so multiple laser scans but only one layer.

Again, to see if your parameters are good with multiple laser scan and when you are satisfied with your multiple laser scan, you start to make some parts. Usually, you make some cubes to test the density and etcetera. It could take a long time when you are starting with very new material.

**Berta Oates**

Thank you. Is there a reason you chose to study non-extrusion-based AM techniques? Do extrusion-based techniques present more complex challenges?

**Flore Villaret**

No, there is no particular reason. Maybe the only reason is that in 3 years of Ph.D., you don't have the time to do all the different processes that exist with additive manufacturing. But, yes, I think we can use the extrusion-based AM technique. I think you think about maybe metal injection molding or something like this. Yes, we can imagine to use such technique to make some gradient materials. With the results that we obtain with the Spark Plasma Sintering and Hot Isostatic Pressing, I think with such technique you will be with close result. I think we will have close result between the extrusion-based technique and the Hot Isostatic Pressing technique.

**Berta Oates**

Thank you. Are there currently methods to certifying parts that are additively manufactured within the nuclear industry?

**Flore Villaret**

It's not an easy question. The answer is naturally we don't have. It's really the beginning, so the only method that we have to certify parts and to be clear we only today to do the parts that are in nuclear reactors and are on non-critical parts. There are parts which are not so subjective to the big scope of the rules [ph] and the link to the nuclear.

But today, the only method that we have to certify parts for the nuclear industry meets with additive manufacturing is you build two parts in the exact same condition. You break one part to show that the second part is okay to be put on a reactor. It's better than nothing. But obviously, we need to improve that because it's okay for small parts but if we want to make some big parts or with high value parts, it won't be satisfying.

**Berta Oates**

Thank you. Has ultrasonic inspection volumetric been conducted on any of these specimens, potentially the joint can be considered as a dissimilar metal weld, similar metal weld plus joint dissimilar metal weld. Here, I am just going to post and then we'll go back through it.

**Flore Villaret**

I didn't perform some non-disruptive technique, but I think it's also a perspective of this work to be able to control with the non-disruptive technique, such parts. Here, at EDF, we have lots of studies going to be able to control and certify parts with the non-destructive techniques.

**Berta Oates**

Thank you, and Dr. van Rooyen has contributed also that there is application of advanced manufacturing parts in nuclear, although it's not yet for safety critical parts which is exactly what you said too, so...

**Flore Villaret**

Yes. I showed at the beginning of the slides, some examples that we have made here at EDF, but I know that the Oak Ridge Institute also made some parts as we do.

**Berta Oates**

Again, thank you and congratulations for your work and your presentation. I think you can tell from the level of engagement, the number of questions and the detail of the questions, how enthusiastic your information has been received. I don't see any additional questions coming in, so I will end it here and thank everyone for participating. As we close out this year, I hope everyone has a healthy and safe holiday season. We look forward to seeing you in the New Year.

Patricia, do you have any closing thoughts?

**Patricia Paviet**

Yes, I wanted again to echo you, to thank Flore for sharing with us your research. You were very engaging with a beautiful presentation, Flore. I wish you well in your career, and I am confident that we have good people for the future of nuclear energy. Nuclear energy is in good hands with people like you. Thank you so much again, Flore. The same as you said, Berta, thank you everyone for following our GIF webinars every month. Wishing you a happy holiday season and we have put webinars for you until

October 2022. I hope you will join again. Thank you, Berta. Thank you, Flore. Thank you, everyone.

**Berta Oates**

Thank you, Patricia. Bye-bye.

**Flore Villaret**

Thank you.

**Patricia Paviet**

Bye-bye, everyone.

**END**

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