

A GENERIC SYSTEM FOR HOMOGENOUS SLS STEEL MATERIALS

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Abstract

A generic approach to a variety of different steel materials for SLS based application has been examined. This approach is based on a base steel alloy powder mixed with a powder blend of much smaller particles. The powder blend is designed to both provide the highest possible density in the powder mass and melting material components for liquid phase sintering to full density. Furthermore, the liquid phase components in the powder blend are composed to both serve as a metallic binder for the green bodies and maintain the possibility to completely dissolve into the base material, forming a homogenous alloy. A powder blend with stainless steel base material was composed and tested and taken through the different process steps. While the formation of green bodies had limited success, to a large extent due to limitations in the process equipment, the sintering and diffusion behaviour showed promising results, both in respect to acquired densities, and homogeneity of the material.

1. Introduction

While conventional polymer based SFF has established a position as an important strategic tool for the manufacturing of prototypes, and is gaining momentum as a competitive direct manufacturing process for parts with complex or unique shapes, a similar development for the metallic applications still seems distant. The most important reasons for this may be found in the combination of the inherent constraints within the different approaches to metallic SFF processes, and the limitations in the selections of materials, available for SFF applications [1]. Therefore, developments in these areas could significantly improve the capacity for new applications of SFF technology. Since steel materials are established for a very wide set of applications, with a long tradition as the engineering materials of choice, most potential users are likely to have steel materials as a major point of reference. Homogenous, SFF applicable steel materials, with similar properties as conventional steels, would therefore not only improve the capacity for SFF manufactured parts, they would also bring the technology closer to the users' point of reference and thereby make it appear less alien to the potential user.

However, for such a material development to have any significant impact, it should preferably be applicable to a SFF system with a wide distribution on the market. This is possible with a procedure beginning with the formation of porous steel powder green bodies by the use of SLS, followed by metallic consolidation and homogenisation by liquid phase sintering in a subsequent furnace process. A generic approach to this procedure, where most variables can be kept constant, -except for those characteristic for the desired outcome, would not only offer SFF products of a homogenous steel material to a wider market, it would also facilitate the development of a much larger number of steel materials for a wide set of applications. (The term "homogenous" is in this text referring to solid non-porous steel alloy, with phase structure corresponding to as-cast material of the specific alloy composition, with respect to heat

treatments. This term is used to mark the difference from the composite or highly segregated materials used in some commercial application of metals SFF.)

The whole procedure, from powder composition, application and processing in the SLS equipment, to the sintering and consolidation during the furnace treatment, is a vast area with a large number of variables, which could be used in many possible different combinations. Considering the endless possible variations, it is hardly possible to scientifically prove that such a material system and procedure is impossible. However, since this hypothesis is concerning technical matters, any successful attempt on any part of the procedure, would indicate that the whole procedure is possible, which is one principal object of this research, Another object is to find which variables are critical to control, and how they affect the outcome of the process. In respect of this, the style of this work will be investigative and argumentative.

2. Background

The formation of parts made of homogenous steel alloy, from SLS, with a subsequent furnace procedure, is a very complex project were a number of variables may be of significant importance. This has been the subject for a series of investigations in the past [2], [3], [4], and [5]. This present work is based on conclusions and acquired knowledge from the preceding investigations as well as the application of established knowledge in conventional powder metallurgy. The approach can be separated into three distinct steps: powder composition, formation of green bodies, and the furnace process, were the limitations and results of each step, affects the conditions for the others.

2.1. Powder composition

If one step should be singled out as being the most critical, it should be the powder composition. Obviously, the powder composition determines the alloy composition of the finished material, but it is also fundamental for every part of each process step, from spreading and application of powder in thin layers, to the sintering into full density and homogenisation of the finished product.

The principal idea of the present process approach, is to combine a base material powder, which's alloy composition will dominate the finished material, with lower melting temperature components, that will serve as a liquid phase during sintering and ideally completely dissolve into the base material, forming the desired homogenous steel alloy. As a most demanding example, a stainless steel, Anval 2205, has been selected as base material for this present investigation, however the principle as such, is generic and could be applied to a vast number of different steel materials.

For liquid phase, previous work has exclusively used copper, [2], [4] and [5], partly for being established in conventional powder metallurgy, as a liquid phase with good sintering properties for iron based alloys, but also because it does improve some desirable properties, such as corrosion resistance, strength and toughness, [6].

However, the amount of copper that can dissolve into the base material is limited, and for successful liquid phase sintering into full density, with a limited amount of liquid phase, it is important to have the powder mass as dense as possible. This has been experienced and

addressed in the past [2], [4] and [5]. It was found that using a suitable combination of size ranges and particle shapes in the correct proportions, will improve the density of the powder layers applied in the SLS equipment, [3]. Since the base material is expected to remain stable, and maintain the geometrical shape of the part during the furnace process, the base material should obviously be the largest fraction and also largest sized particles in the powder blend. Additional components, such as the melting material, should, if possible be kept at particle sizes that will fit into the voids between the larger particles.

This approach has been tried with $-44\mu\text{m}$ Anval 2205 powder, copper powder of the smallest particle size available, and with an additional amount of small particle size carbonyl iron powder for further densification, [5]. However the high inner friction, and the unexpected unfavourable size range relation between base powder and copper in the powder mass gave the powder blend poor particle packing properties. This, in combination with the low fraction of melting material for the liquid phase sintering (8%), made it impossible to achieve full density in the material. Therefore has this present investigation changed the size range for the base material to $44\mu\text{m} - 75\mu\text{m}$, and added a second melting material: ferrophosphorus. **Fig.1. & Tab.1, Fig.2. Fig.3. & Tab.2, Fig.4. & Tab.3.**

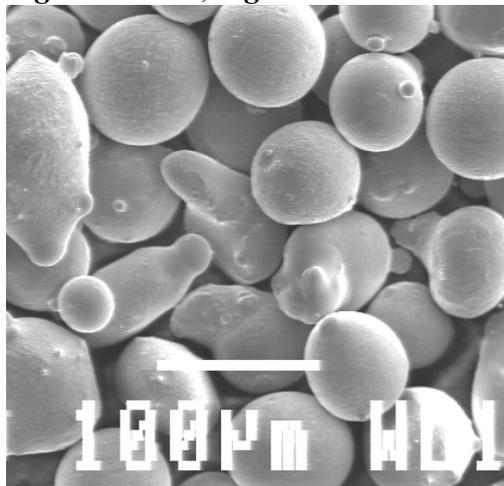


Fig.1. Anval2205. A stainless steel material, with the characteristic smooth shape of gas atomised metal powder. This alloy has been selected as a challenging material with interesting properties. Size analysis: ASTM-E11 sieve, 99 % < $-75\mu\text{m}$, 9 % < $-45\mu\text{m}$.

C	Si	Mn	P	S	Cr	Ni	
0.019	0.70	1.17	0.017	0.002	22.3	5.17	

Mo	Nb	Cu	Co	V	W	N	O
3.02	0.00	0.16	0.03	0.07	0.02	0.19	165

Table 1. The chemical composition of the stainless steel base material, Anval 2205, O in ppm.

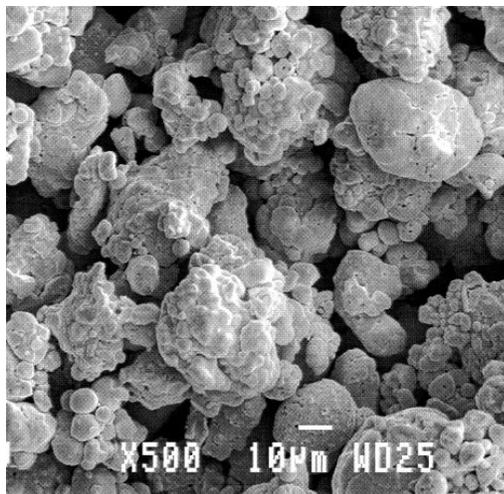


Fig.2. Copper powder, 99.9% Cu. According to vendor $1-5\mu\text{m}$ spherical shape, which in reality obviously is not the case. This discrepancy is however most likely due to the method of measuring and calculating size. In many cases are such small sized particle's sized estimated by a measured value for particle surface that is recalculated to volume and diameter assuming spherical shape. For a sponge like powder such as this, this method would indicate a much smaller particles size than really is the case. For this investigation this defect will not disturb the size range composition, however, it will limit the density improvement.

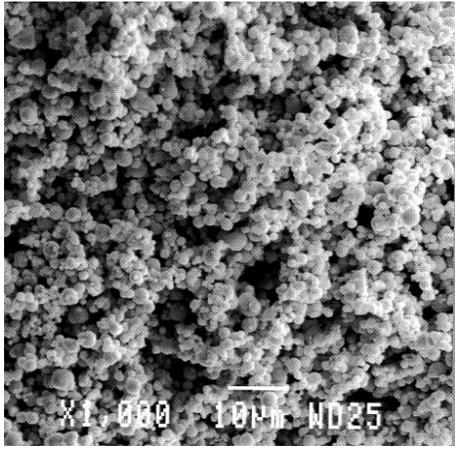


Fig3. Carbonyl Iron SM: almost pure iron, with the spherical shape typical of carbonyl metal powder. In this size range (2-8 μm), the interparticle friction is associated with van der Waal's and electrostatic forces, and is thus increasingly independent of particle shape. Nevertheless, the spherical shape still is the best possible considering the achievable packing properties.

Fe	C	O	N
Min. 99.0	Max. 0.2	Max. 0.8	Max. 0.1

Table 2. The chemical composition of the filler material, Carbonyl SM

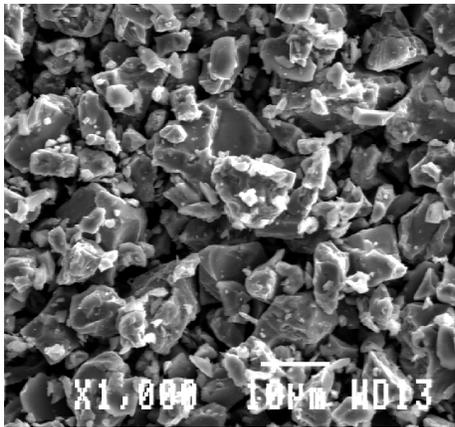


Fig.4. Ferrophosphorus powder, coarse and uneven. Particles. This has normally a negative effect on powder packing properties. However at this size range are these properties dominated by the powder mass' internal friction, associated with van der Waal's and electrostatic forces. For this investigation, surface smoothness of ferrophosphorus particles will be of minor importance.

Fe	C	Si	Cr	Mn	P	Ti
Base	0.78	Max. 0.50	Max. 0.50	Max 0.50	15.6	Max 1.50

Table 3. The chemical composition of ferrophosphorus powder. Physical properties (Sympatec): 10% below 2.61 μm , 50% below 8.25 μm , 90% below 15.23 μm

Phosphorus, P, is a well-known additive to powder compositions to improve sintering properties by liquid phase sintering, though other positive side effects, such as improved tensile strength, elongation and hardness, with small additions of P, has been reported from conventional powder metallurgy, [7]. The best results were acquired with additions of 0.75 % - 0.80% P, and the added amount of ferrophosphorus will therefore be adjusted to yield this content of P to the finished material.

2.2. Formation of green bodies

This process is expected to be capable to deliver hollow parts and geometries with deep cavities, and since the part in all cases will be subjected to a significant shrinkage during liquid phase sintering, a supportive powder during the furnace process is not desirable. Therefore, organic binders, requiring a supportive powder during the binder burn out and the early stages of sintering, should be avoided. This issue has been addressed in previous work, [4] and [5], and it has been found that despite the low absorbance of energy from a carbon dioxide laser, copper can be melted during SLS processing and thus work as a metallic binder. The green bodies formed are however quite brittle, with especially weak bonds between the layers. On the other hand, in this present investigation, there is possibility that the second melting phase, ferrophosphorus, also could melt at process conditions and improve this situation.

Since copper has such low absorbance for energy transmitted by carbon dioxide lasers [8], it is more likely that it is secondary heating that melts the copper. The laser energy is absorbed by the iron-based particles, which in turn heats the adjoining copper particles until melting and bonding occurs. If so, the increased particle size will require more energy to raise the temperature of the base material particles to the melting point of copper (1084.5 °C) and even more for ferrophosphorus, (melting point 1168.0 °C), if the same heating mechanism is active as in copper. Since the equipment available for this investigation is a rather old Sinterstation 2000, 50 W CO₂ laser, with a measured maximum output of 41.5 W, this may be a crucial limiting factor for the formation of green bodies. However, the formation of perfect green bodies is not the primary object of this investigation, but the establishment of a generic principle for a series of homogenous steel materials that could be achieved from a procedure consisting of SLS and a subsequent furnace process. For this object, sintering and homogenisation behaviour is more vital, and it is sufficient to show that green bodies *can* be formed by SLS, while quality is of less importance at this stage.

2.3. Furnace process conditions

The feasibility, as well as process time and temperatures for the furnace procedure, in order to achieve a homogenous, liquid phase sintered steel material, are determined by some important boundary conditions. First of all, the process parameters must allow the liquid components (here: copper and ferrophosphorus) in the powder composition to melt and sinter the material to full density. Second, the process time and temperature must be sufficient for the liquid material to completely dissolve into the base material, as well as allowing the alloy components from the base steel to homogeneously be distributed within whole part, -both by means of diffusion. Third, since the risk of evaporation of alloy components from the material during the vacuum process is controlled by the combined effect of temperature and pressure, there is an upper limit on the acceptable temperatures, -or a lower limit for pressures, at process conditions. Meanwhile the pressure in the process chamber must be kept low enough to allow desoxidation of the material during the process. Fourth, the processing time must be as short as possible, partly to avoid unnecessary grain growth, but also to keep the process times at a competitive level.

However, this complex situation can be simplified by applying standard settings for vacuum pressure, and thus leave time and temperatures as the process controlling variables. Further simplifications can be made by application of conclusions from previous work. It has been found that at standard vacuum settings, temperatures above 1200 °C will cause evaporation of chromium from stainless steel powder, (1233.6 °C for 22.4% Cr) [5]. Therefore 1200 °C can be considered a maximum process temperature, which, in combination with vacuum, will allow the desoxidation reaction between carbon present in the powder blend, and oxide on the particles' surfaces. This leaves process time as the critical variable to control.

Since the copper's only means for dissolving into the steel material under these process conditions is by self-diffusion, and the longest predictable diffusion distance for any material under these circumstances is from the surface to the centre of the largest particles, the diffusion of copper through the base material will be the determining factor of the process times. This simplification may overlook the closure of large pores, but the occurrence of those, as well as their size and distribution, is not entirely predictable, and must be experienced experimentally.

The previously mentioned investigation, [5], also found that 4 h would be sufficient process time for 8 % copper to completely dissolve into -44 μm base material steel powder. However, in this present investigation has the size range for the base material been changed the to 44 μm - 75 μm , and ferrophosphorus has been added as a second melting material. The increased particle diameter gives an increased maximum diffusion distance, which, reasonably, would increase the required process times beyond practical and competitive limits. However, that investigation and the following estimation of process time are based on the assumption of the atomic configuration of γ -iron, which would normally be the phase present for most steels at process temperatures, especially since the added copper is γ -stabilising. But, in this case, ferrophosphorus is added as a second melting material, and this could vitally change the conditions for the diffusion of copper and other alloying components.

Phosphorus is known to have a powerful stabilising effect for α -phase and, compared to for example copper, a rapid diffusion behaviour in both α -, and γ -phase. Therefore, the hypothesis is, that the phosphorus will diffuse into the base material of γ -phase configuration, ahead of copper and other significant alloying components, and thus cause a phase transformation to α -phase. Since the atomic configuration of α -phase steel allows much higher rates of diffusion for copper than γ -phase, this would probably make it possible to achieve homogeneous distribution of alloying- and former liquid components in the finished steel material within reasonable process times. This, in combination with the additional amount of liquid phase and increased powder packing densities could make it possible to achieve a homogenous steel material with no, or very low porosity, by SLS and a subsequent furnace processing.

2.4. Calculations

The time to dissolve the copper into the base material is controlled by the fractional saturation of copper in the base material particles. Darken & Gurry [9] gives us the relationship:

For diffusion into spherical shape: $Dt/L^2 = -0.05043 - 0.2333 \log(1 - F)$, [Eq. 1]

Diffusion constant, $D = D\gamma_{Cu}^{Fe} = 1.8 \exp\left(-295 \text{kJmol}^{-1}/RT\right) \text{m}^2/\text{s}$, [Eq. 2], [10].

Fractional saturation, $F = \frac{C_m - C_0}{C_s - C_0}$, [Eq. 3], and L is the diffusion distance, here the radius of the

base material particles. From previous work [5], we know that at 1200 C, 8% copper can theoretically be completely dissolved in spherical -44 μm steel powder, in 3.85 hours. For reasons previously mentioned, a particle size range of 44 μm - 75 μm has been used for this present investigation. With 75 μm powder particles, the diffusion distance is 0.00375 cm. Added copper, $C_m = 8\%$, at 1200 C saturation of copper is $C_s = 9\%$, which gives $F = 0.889$. Applied to [Eq. 1], $Dt/L^2 = 0.17$. At 1200 C is $D\gamma_{Cu}^{Fe} = 6.21 \cdot 10^{-11} \Rightarrow t = 369.42$ h, which is too long. However, with phosphorous induced phase transformation, diffusion is expected to take place in α -phase where,

$D = D\alpha_{Cu}^{Fe} = 8.6 \exp\left(-250 \text{kJmol}^{-1}/RT\right) \text{m}^2/\text{s}$ [Eq. 4], [10]. Under these conditions

$D\alpha_{Cu}^{Fe} = 1.17 \cdot 10^{-8} \Rightarrow t = 204.32$ s (!), which is much more reasonable.

However, these ideal conditions cannot be fully expected under real process conditions. For example, if the material has sintered to full density, the particles will not be spherical during the whole dissolving process. Complete homogeneity concerning pore size and orientation, or particle distribution within the green body can neither be expected. Furthermore, the liquid phase will be a solution melted copper and ferrophosphorus, which means that the concentration of the different elements will vary during the process, and during the diffusion of copper, the balance between α -, and γ -phase in the outer perimeter of each particle is difficult to predict, and therefore also the real diffusion rate. Considerable longer process times will therefore be used for this investigation, however the calculations above demonstrates that complete dissolving and diffusion to homogeneity is not unreasonable to expect with this material composition and process conditions.

3. Experimental

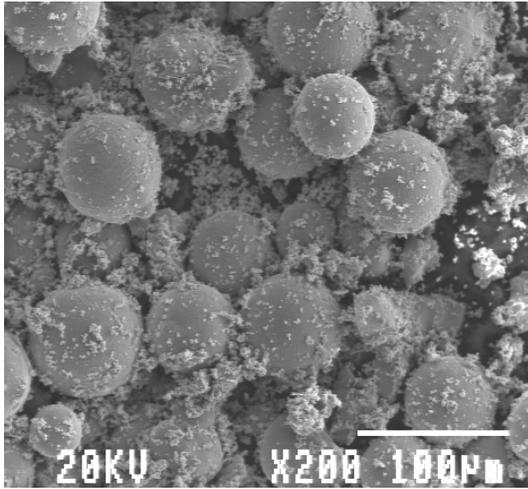
The experimental part of this investigation follows the principles outlined in the background section. The powder blend is composed to yield maximum applied powder density using the selected powders. Green bodies are formed using SLS equipment, and processed to full density in a vacuum furnace. Samples examined regarding porosity and have been further analysed regarding alloy composition and distribution using SEM equipment at the Department of Materials and Engineering, KTH.

3.1. Powder composition

The different selected powder materials have to be combined in the correct proportions to yield the expected results. The amounts of the different liquid components is determined by their respective solubility in the base material, and possible desirable effects on the material properties of the final alloy. Copper has a good solubility in iron-based alloys up to around 8 %, at process conditions, which settles the share of copper. Phosphorus has been reported to have most positive effect on material properties at 0.8 % P. The added ferrophosphorus contains 15.6 % P, which makes an addition of 5.12 % yield the desirable amount of P in the final material. This combination would give a liquid share of 13.12 %, which is slightly less than conventional, [11]. However if the experiment should indicate that the liquid share is too small for pore closure, it is possible to increase the addition of ferrophosphorus.

Besides getting the right amount of liquid phase, it is also important have the highest density possible in the powder mass. Since the amount of small sized particles for the liquid phase has been determined by other boundary conditions, the remaining means to control this issue is the combination of large particle base material, Anval 2205, and a fill material powder with much smaller particle size, in this case Carbonyl Iron SM.

The suitable proportions was found experimentally using the procedure described in [3], with the amounts of copper and ferrophosphorus kept constant at 8 % respective 5.12 %. The final powder composition can be seen in *Tab.4. Fig.5*, showing the powder blend gives an impression of particle size proportions and packing behaviour. The density of the applied powder layers in the SLS equipment was measured by the procedure described in [3], and was found to range between 65.8 % and 67.4, which seems promising for the following process steps.



Anval 2205	Carbonyl SM	Cu	Fe ₃ P
75.88 %	11.00 %	8.00 %	5.12 %

Table 4. The composition of the powder blend.

Fig.5. The powder composition after mixing.

3.2. Formation of green bodies

The determined powder composition was exposed to SLS processing for several attempts with successively increased laser power and decreased scanning speeds before any usable green bodies could be formed. This occurred at the very limit of machine capacity. Apparently, the assumption concerning the need for increased energy due to the increase in base material particle size was correct. Still, green bodies were formed, however of poor strength, with very weak bonds and high porosity between the layers, and with a strong tendency for curling of the layers. It is a likely assumption that only copper melted to serve as a binder, and that this situation could improve, -in terms of improving green strength by melting the of ferrophosphorus, with the use of a more powerful laser, such as the 100 W laser common in more modern versions of SLS equipment from DTM/3Dsystems, or the even stronger lasers used by EOS.

3.3. Furnace processing

Samples were sintered in a conventional vacuum furnace, in accordance with conditions described in the calculations section, and the principles described in [12]. The parts were placed on an alumina bed, in a graphite crucible, with no supportive powder and loaded into the furnace. The temperature was raised to 1200 °C at a rate of 200 °C/h. held at that temperature for 4 h, when the temperature was lowered at the same rate.

4. Results and analysis

The samples had sintered to solid objects and undergone some shrinkage, **Fig.6**, notable is the fact that the samples with less successful layer adhesion have had less shrinkage in X – Y –direction, instead, the more separated layers have had a predominant shrinkage in Z –direction, whereas the samples with more successful adhesion between the layers have shrunk more equally in all three dimensions. Furthermore, some sagging of large overhangs can be noted. Looking deeper into the material we find that there are some remaining pores, predominantly oriented in between powder layers, **Fig.7**. However none of these imperfections are unexpected, and could probably be traced to the less than perfect conditions during the formation of green bodies.

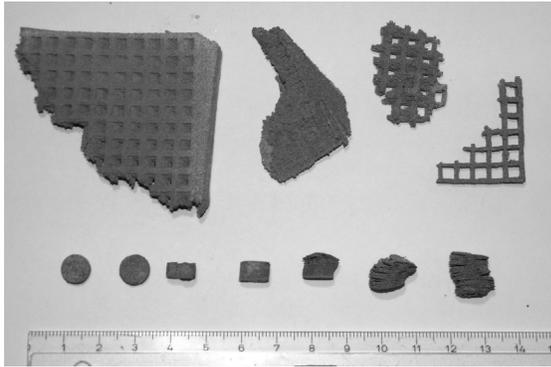


Fig.6. Sintered samples. Shrinkage and some sagging of overhangs are evident. Especially notable is the shrinkage of the cylindrical samples in the bottom line. The same STL file and laser power was used for all of them, however with different scanning speeds.

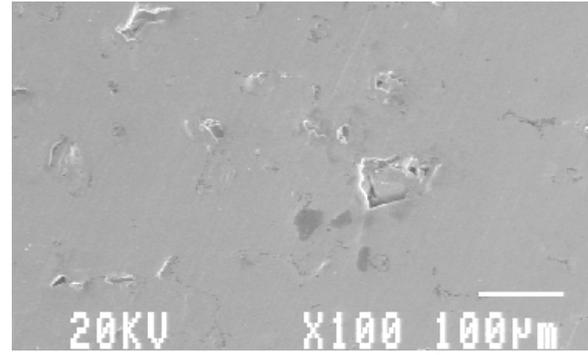


Fig.7. Pore structure, close to the boundary between two layers. The porosity increases with decreasing layer adhesion due to increased curling of the layers at lower laser scanning speed

More interesting, still, is the analysis of material alloy and distribution of alloy elements in the material. It shows that complete homogeneity has not been reached, which is quite reasonable, complete homogeneity hardly exist even in conventional steels. Certain elements naturally concentrate into certain phases, and nothing else could be expected under these circumstances. But the distribution analysis as shown in **Fig.8** and **Fig.9**, displays encouraging tendencies.

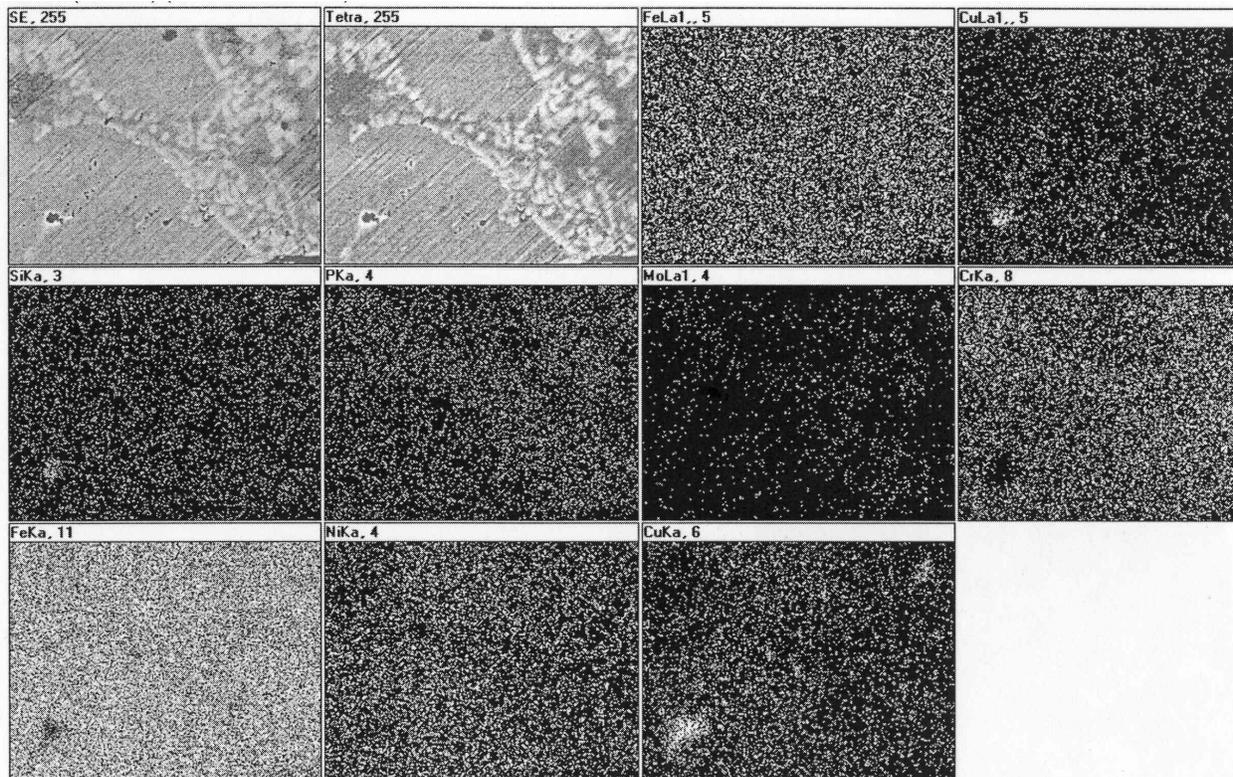


Fig.8. First distribution analysis of important elements covering an area of 58.9 x 44.6 μm . Fairly even distribution throughout the part, however complete homogeneity has not been reached. As could be expected have some elements, such as Mo, had the tendency to concentrate to certain phases as is normal in conventional steels. Also interesting is the area of Cu and Si concentration in the lower left side corner. Apparently has this been an area where larger amounts of liquid phase has been gathered than could be dissolved in the surrounding base material.

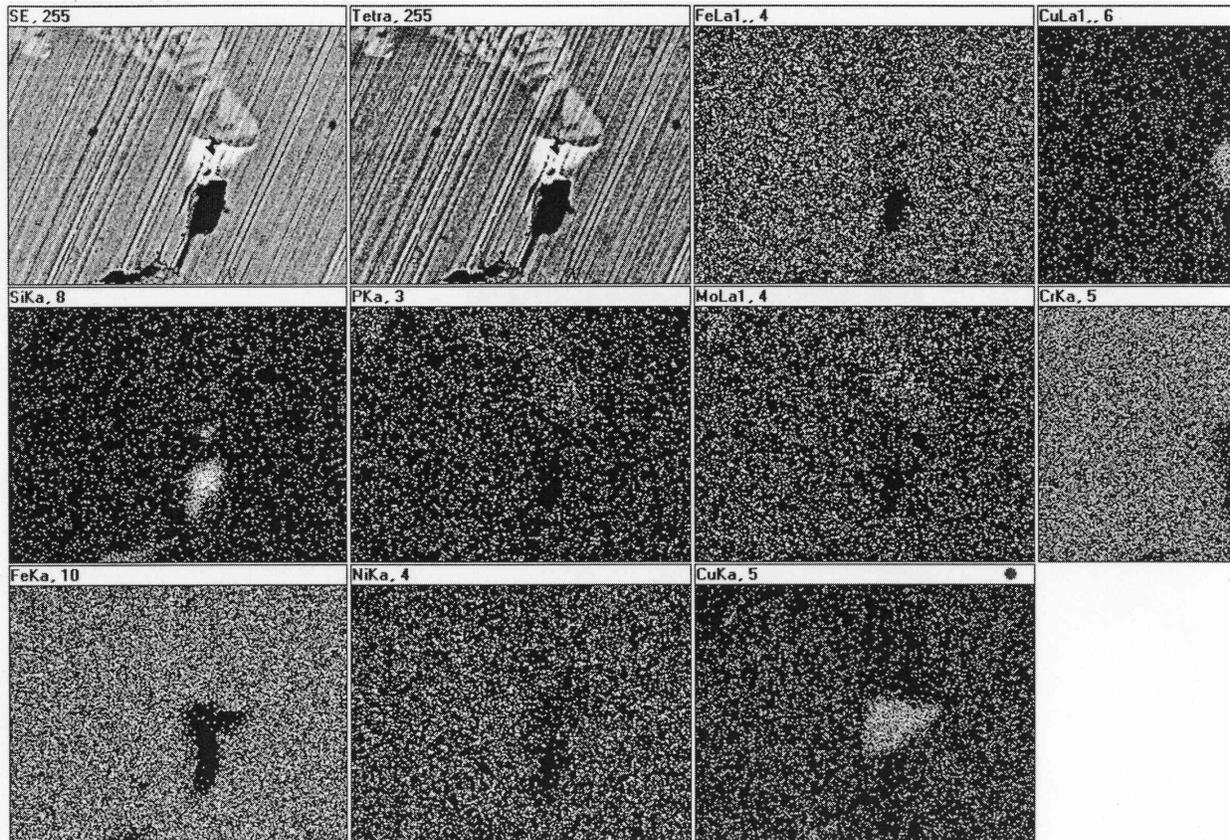


Fig.9. The second distribution analysis of important elements covering an area of $51.0 \times 42.5 \mu\text{m}$. The tendencies from previous analysis appear to be confirmed. There are some areas where Cu and Si have been enriched, while P and Mo have been concentrated to certain phases.

From an overall view, all elements are fairly even distributed over both areas analysed. There are however some points where certain elements (for example Cu & Si) are especially concentrated, and other elements, for example iron, is sparse. This could probably be traced back to the liquid phase during sintering. Silicon is highly soluble in both liquid copper and iron (such as in ferrophosphorus), and since phosphorus has the higher diffusion rate, iron will leave the liquid phase ahead of copper, thus the dissolved silicon will concentrate to the liquid copper. As pores are closing during the sintering process, remaining liquid material, in this case copper, will concentrate to the remaining larger pores. With the larger amounts of copper amassed in a small region, it is hardly surprising that all copper will not dissolve into the surrounding material within the limited process time.

5. Discussion and conclusions

The primary aim for this investigation was to explore a possible generic principle to build parts of homogenous steel, using SLS equipment in combination with a subsequent furnace procedure. Despite the obvious shortcomings of the parts manufactured, it is not unreasonable to say that this possibility has been confirmed. Solid pieces of steel were formed from green parts made in the SLS machine, and the analysis of the distribution of the principal alloy elements shows, with some exceptions, a reasonable even dispersion.

However the material selected for this investigation was a stainless steel, and apart from the most general material properties, such as tensile strength, hardness, and others, the corrosion resistance of the new alloy still has to be determined. This could be problematic, however, since there still are some areas with significant porosity or with excess concentration of, for example, copper. How much this would influence the corrosion resistance by crevice- or galvanic corrosion is unclear, but any measurements on the available pieces would not be conclusive either. As mentioned earlier, the areas with excess copper concentration probably originates in large pores, and since the porosity is primarily oriented between the applied powder layers, these could thus in many cases be deduced to poor adhesion of powder layers. Small pores could be addressed by increasing the fraction of liquid phase in the powder blend, but the large pores, which could end up filled with remaining copper, are more difficult and the best would be if they could be avoided altogether. One likely way to at least decrease their frequency could be to improve the adhesion between the layers.

The most obvious method to improve layer adhesion would be to increase the laser power. A more powerful laser, such as those installed in more modern SLS machines on the market by DTM/3D Systems and EOS would penetrate deeper into the powder layer and thereby increase the melting of material in between the layers. Furthermore, the higher laser effect would enable the melting of the ferrophosphorus during SLS, thus increasing the metal bonding and thereby further improve the quality of the green bodies. On the other hand more extensive metallic bonding will also increase the temperature induced tensions in the green bodies, though these tensions will be relieved during the furnace process. If needed for complete pore closure, the fraction of liquid phase (here the ferrophosphorus) could be increased. However, a less desirable effect, of increased fraction liquid phase would most likely be an increase of the small sagging observed on the sintered parts, and thus loss of part geometry. On the other hand this behaviour could probably be moderated by stronger metallic bonds in the green bodies that comes with an increase in laser power.

The full process of making solid homogenous steel objects from an SLS approach could not be verified by this investigation. However, the acquired results concerning sintering and diffusion are encouraging, and while the observations of the SLS behaviour are challenging, they do not contradict this possibility.

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