

Proposal to KME

Applying organisation	Division of Materials Microstructure, Department of Applied Physics, Chalmers University of Technology
Organization number	556479-5598
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Co-applicants	Prof. Hans-Olof Andréén (Chalmers)
Project	Continuation project of KME 510

Design of a new generation of 12% chromium steels

Summary

Z-phase strengthening is the only possible solution today to achieve good creep and corrosion resistance at 650°C for 9–12% Cr steels. Research on this new generation of steels was initiated in Denmark and Sweden (DTU, Chalmers and Siemens). The proposed project is a renewal of our successful KME-510 project, where we proved that the novel concept of using Z-phase as a strengthening phase is feasible. These steels will enable higher steam temperatures and pressures, and thermal efficiency of power plants. They also open the possibility to replace austenitic steels in superheaters with martensitic steels, thus improving the operating flexibility. In this project, we plan to continue the work of developing Z-phase strengthened 12% Cr steels. The focus is to fine tune the chemical composition and heat treatment to further improve long-term creep resistance, and to make them more economically viable. This project will co-finance a Ph.D student (licentiate in 2015, and Ph.D in 2018).

Sammanfattning

För närvarande är Z-fasförstärkning den enda möjliga lösningen för att erhålla såväl högt krypmotstånd som god korrosionsbeständighet vid 650°C hos 9–12% kromstål. Forskningen om denna nya stål initierades i Danmark och Sverige (DTU, Chalmers och Siemens). Detta projekt är en fortsättning på vårt framgångsrika projekt KME-510, i vilket vi visade att nya konceptet att använda Z-fas som partikelhårdande fas är praktiskt möjligt. Dessa stål kommer att möjliggöra högre ångtemperaturer och ångtryck, vilket medger högre termisk verkningsgrad i ångkraftverk. De öppnar också möjligheten att ersätta austenitiska stål i överhettare med martensitiska, vilket förbättrar driftflexibiliteten. I detta projekt önskar vi fortsätta arbetet att utveckla denna nya stål. Fokus ligger på finjustering av sammansättningen och värmebehandlingen för att ytterligare förbättra kryphållfastheten och samtidigt göra dem billigare. Projektet ska medfinansiera en doktorand, licentiatexamen 2015 och disputation 2018.

Motivation

Through this project we would like to gain knowledge on creep of 12% Cr steels for more advanced steam parameters (higher steam pressure and temperature). It matches well with one of the goals set by the “**materialteknisk utveckling**” in the KME programme:

- Genomföra studier i labbskala för att utvärdera mekanismer som har största påverkan på materialens mekaniska livslängd i relation till nya krav på materialen vid högre ångtryck och ångtemperaturer.

In a long-term, the proposed project can also contribute to the following goals set by the “**Process- och anläggningsteknisk utveckling**” in the KME programme

- Undersökt möjligheter och hinder för hur anläggningar i Sverige kan nå högre ångdata motsvarande långsiktiga visionen om 3-4 procentenheter högre elverkningsgrad än bästa teknik för givet bränsle idag.
- Utvärderat olika materials mekaniska egenskaper och livslängd i relation till nya krav på materialen för effektivare elproduktion (höjda tryck och temperaturer).

Energy relevance

The research can lead to a new generation of steels, i.e. Z-phase strengthened steels, which fulfil basic requirements on mechanical properties and combine good long-term corrosion and creep resistance. It is a promising candidate material for steam turbine rotors and turbine blades, which is directly beneficial for Siemens Turbinmachinary AB for producing a new generation of steam turbines applied in a more advanced steam parameter of 650°C/325bar (vs. today's 600°C/280 bar). Furthermore, it can also be used in superheaters in biomass fired power plants for the nearest future, where the steam temperature would be enhanced from today's 540 °C to 580 °C. After proper coating of the surface at the fireside, these steels can survive the harsh chemical environment in the bio-fuel boilers.

In addition, martensitic steels provide several advantages compared to austenitic steels, which are widely used in superheaters today. Martensitic steels offer both satisfying mechanical properties and steam side corrosion resistance. They are at least two or three times cheaper than austenitic stainless steels or ten times cheaper than nickel based superalloys. More importantly, martensitic steels have higher thermal conductivity and lower thermal expansion. Their superior thermal properties allow a much quicker start up, and ramp down procedure in power plants without causing severe thermal stress in the superheaters. Flexibility in power plant operation is particularly valued in the future in order to accommodate the huge power fluctuations from renewable energy sources, like solar and wind energy.

Industrial relevance

Through the close collaboration between Chalmers, Siemens Industrial Turbomachinery, and the Technical University of Denmark (DTU), this project can further enhance the understanding of an important class of steels (i.e. 9–12% Cr steels) for the power generation industry. Within the project, knowledge is efficiently transferred between the two parties, which can enhance the competence of them all. Also educating graduate students within the project can add in fresh competence to the Swedish industry. In particular, on the Siemens

side, the deep insights into the steels enabled by a comprehensive understanding of the microstructure can be readily used in developing new products using the new steels. Some specific knowledge, like the influence of heat treatments (a very industrial relevant process) on materials microstructure is beneficial also for their future development programmes.

General applicability

The new 12% Cr martensitic steels can be used not only in superheater tubes, given the good physical properties they possess, they can also be used as turbine blades and turbine rotors, for e.g. concentrated solar power plants or steam power plants that burn other types of fuels and with steam temperatures reaching 650 °C. For countries that are still forced to build new fossil fired power plants the new steels can lead to a decrease in CO₂ emissions by 10% compared to state of the art (650 °C vs. 600 °C, 50 vs. 45 % efficiency).

News value with the project

Z-phase strengthening is the only way to gain enhanced corrosion resistance without losing creep properties at the same time. Our goal is to push the operating temperature up to 650 °C, which is believed to be the upper limit for 9–12% Cr steels. None of the present 9–12% Cr steels fulfil the requirements concerning both corrosion and creep.

Implementation of results

Our close collaboration with Siemens ensures that promising trial steels are further developed. In addition, our participation in the EU FP7 programme Z-Ultra gives us access to welding development (Paton Institute, Kiev), up scaling (forgings at Saarschmiede), and field tests (superheater tubes in Kievenergo plants).

Background

Previous and on-going work on new 12% Cr steels at Chalmers

Work at Chalmers on the creep behaviour of modified 12% Cr steels started in 1990 (Prof. Andrén). Since many years there has been a close collaboration with DTU (Prof. John Hald), and Siemens Industrial Turbomachinery, the turbine maker in Finspång (Lennart Johansson and Helena Oskarsson).

In 2010, Prof. Hald et al. proposed a novel concept of designing a new generation of 12% Cr steels using Z-phase as a strengthening instead of a detrimental phase. Since then intensive research has been carried out in order to prove this design concept and to optimize the design at Chalmers, DTU and Siemens. Financial support was received from KME (2011-2014, 1000 kSEK), Energimyndigheten (via “VR energiriktad grundforskning”, 2011-2013, 2,565 kSEK), Research Foundation of VGB (2011-2013, 75 kEUR), and the EU-FP7 (2013-2015, 388 kEUR for Chalmers out of a total budget of 2,548 kEUR).

The proposed project is to support renewed research activities on the new 12% Cr steels, in order to address one of the grand challenges faced by the materials used in future biomass steam power plants – the dilemma between improving corrosion resistance and creep strength.

Challenges for 12% Cr steels for the future biomass-fired steam power plants

Combustion of biomass has great potential to combat CO₂ emission. This process is often labelled as net carbon neutral, since the biomass has to bind up CO₂ during the growth. Compared to the state of the art fossil-fired power plants, biomass-fired plants are run at a much lower temperature (540 °C vs. 620 °C), due to severe corrosion attack to the fireside of the superheaters.

For these power plants, focus is put on enhancing plant efficiency, mainly through increasing operating steam temperature and pressure. For biomass-fired plants, the nearest goal is to increase the steam temperature to ~580 °C, which means that the temperature of the metal in the superheater is ~630–650 °C. At the same time, we must keep the construction and maintenance cost as low as possible, in order to make energy produced in these plants attractive to energy consumers. Furthermore, for the future these plants must possess greater operation flexibility, allowing quick start up and ramp down circles, in order to accommodate the fluctuation nature of other renewable energy sources, like solar and wind energy, and to load-balance the electric grid.

Tempered martensitic 9–12% Cr steels offer an optimal combination of the critical properties, i.e. creep strength, corrosion resistance at the steam-side, and thermal properties, at a relatively low cost [1]. Therefore, with a proper coating at the fireside, a new generation of 9–12% Cr steels, which possess high creep strength at 650 °C, can provide reliable and relatively cheap superheaters for the biomass fired power plants.

Our goal is to push the operating temperature up to 650 °C, which is believed to be the upper limit for 9–12% Cr steels. None of the present 9–12% Cr steels fulfil the requirements concerning both creep and corrosion. There have been several recent attempts to achieve the goal. Nevertheless, all of them turned out to be failures.

The reason behind this lies in the phase transformation of an important family of precipitates concerning creep, MN (M = V, Nb), to thermodynamically more stable Z- phase, CrMN, during creep. A novel steel design concept, which makes use of Z-phase to strengthen the steels, has a great potential to realize the goal by improving both creep strength and corrosion resistance. These are discussed in detail in the following sections.

Microstructure of conventional 9–12% Cr steels and creep resistance

In the technically interesting stress and temperature range for tempered martensitic steels, the main creep mechanism is dislocation creep. Therefore, the ways to retard or delay dislocation glide and climb are helpful to improve creep resistance [2]. The most important strengthening mechanism is precipitation hardening [3].

The size and number density of the precipitates at the initial stage, as well as their stability against coarsening during exposure to high temperatures play a vital role in creep. Among the main precipitates, the carbide M₂₃C₆ (M = Fe, Cr, Mo), nitride MN (M = V, Nb, Cr), and the intermetallic Laves phase Fe₂M (M = Mo, W) are beneficial, while the complex nitride Z-phase (Cr,Fe)MN (M = V, Nb, Ta) is normally considered detrimental.

M₂₃C₆ precipitates are the major strengthening particles for the first generation of 9–12% chromium steels like X20CrMoV121. They are mainly located at the prior austenite grain boundaries and lath boundaries, with a typical size of 100 nm after tempering and a few

hundred nanometres after prolonged exposure. At late 1970's, researchers at Oak Ridge National Laboratory succeeded in obtaining very finely distributed MN precipitates by adding small amounts of V or Nb and N into the alloy - P91. And a few years later, another commercial steel P92, which has similar chemical composition as P91 but also contains additions of W and B, was launched. P91 and P92 are mainly based on particle strengthening with MN and $M_{23}C_6$. Compared to $M_{23}C_6$, MN has a much smaller particle size, usually at the range of 20 nm after tempering, and higher number density. More significantly they are very stable against coarsening in P91 and P92 [4]. This makes P92 twice as strong as the old X20CrMoV121 at 600 °C.

Z-phase formation and strength breakdown

P92 contains only 9 wt.% Cr, which is too low in terms of corrosion resistance at 650 °C. It has been established that at high temperatures the corrosion resistance of a steel depends on its Cr content; the higher the Cr content, the better the corrosion resistance [5]. Therefore there have been several recent trials aiming at improving corrosion properties by adding more Cr (11–12% vs. 9% in P92). Nevertheless, all of them turned out to be failures. Although they showed better creep resistance at 650 °C than older steels until ~10,000 hours, they then suffered a dramatic loss of creep strength [6].

It has now been widely accepted that the mechanisms behind the dramatic breakdown were mainly attributed to the precipitation of a complex nitride known as Z-phase ((Cr,Fe)(Nb,V)N) during creep. Many small MN precipitates are dissolved to form large Z-phase particles, which are thermodynamically more stable at this temperature but give very little contribution to creep strength [7]. Z-phase formation in a number of 9–12% Cr steels, which rely mainly on MN for strengthening, has been systematically studied. It has been shown that a Cr content above 10.5% strongly accelerates Z-phase formation. In contrast, steels with 9% Cr or below are largely unaffected by the Z-phase precipitation up to 100,000 hours at 600–650 °C [8].

New 12% Cr martensitic steels with Z-phase as strengthening precipitates

Z-phase precipitates were credited as beneficial for strengthening in creep resistant austenitic steels containing Nb, since they precipitate quickly among the first appearing precipitates and as fine densely distributed particles.

Our collaborators from DTU proposed a new alloy design concept, which makes use of Z-phase as strengthening dispersion for 9–12% Cr steels, instead of MN. The new steels are supposed to be strengthened by fine Z-phase precipitates, which are formed during heat treatment or at the early stage of service. It is expected that the steels possess both good creep and corrosion resistance, which is attributed to dense distribution of stable and fine Z-phase precipitates and a high Cr content (12 at.%) [8].

Goal

We will perform systematic research in order to gain a comprehensive understanding on the creep mechanisms of these new 12% Cr steels. We will provide fundamentally important knowledge on the sophisticated process-microstructure-properties relationships, which can be fed into the improvement of the new steels so that the new steels with optimal combination of corrosion and mechanical properties can be exploited for use in thermal power plants. Success of this type of steels could enable the construction of low cost superheaters in

biomass-fired power plants with steam parameters of up to 580 °C entirely based on these martensitic-ferritic steels rather than the costly austenitic steels.

Within the frame of this project we plan to:

- Based on the results obtained from the previous KME project and the parallel EU project, design three new test steels with fine-tuned chemical composition, aiming for improved creep resistance compared to the ones that have been designed and investigated in the previous KME 510 project;
- Optimize heat treatment conditions for test steels;
- Perform mechanical and creep testing on promising test steels with optimized heat treatment;
- Understand the sophisticated effects of carbon addition on the precipitation reaction sequences in the Z-phase strengthened steels;
- Understand effects of small addition of B on the Z-phase strengthened steels;
- Understand special effects of Ta in combination with Nb;
- Understand evolution of microstructure and its influence on the creep mechanisms.

Within the framework of this project a Ph.D. student (Masoud Rashidi) will also be educated:

- Licentiate planned in October 2015.
- Ph.D planned in April 2018.

Project plan

In the proposed project we plan to make systematic research on some of the fundamental issues for Z-phase strengthened steels, like the nucleation process of Z-phase precipitates, the microstructure evolution of the steels under the influence of different alloy addition, such as Ta in combination of Nb; various level of C addition; and their subsequent effects on the creep strength. Siemens will perform heat treatment and mechanical testing including tensile strength, hardness, impact toughness and long-term creep testing.

We start with a short summary of our previous KME 510 project, followed by a detailed project description and time plan.

A short summary of KME-510

In the KME-510 project (2011–2014), we have proved that the concept of using fine and densely distributed Z-phase to strengthen 12% Cr steel is feasible. Despite their much higher

chromium content the proof-of-concept alloys showed excellent creep resistance, comparable with benchmark alloys – P91 and P92, two of the most widely used commercial 9–12% Cr steels today (Figure 1).

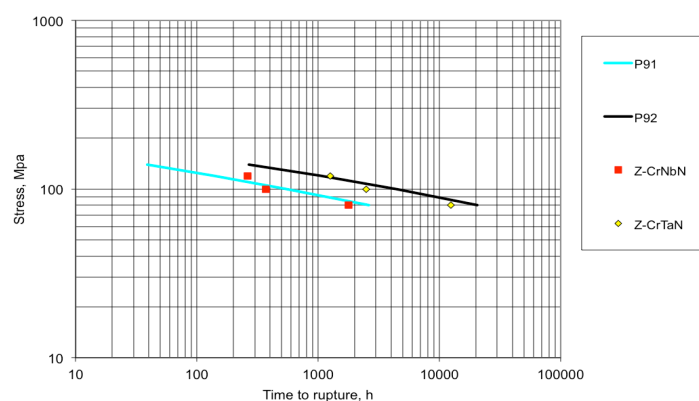


Figure 1. Creep rupture results at 650 °C for the new Z-phase strengthened steels: the CrTaN-containing and the CrNbN-containing steel, in comparison with two benchmark commercial steels, P91 and P92.

Further fine tuning of the alloy chemical composition was performed to address two issues: firstly to improve the room temperature impact toughness of the proof-of-concept alloy; secondly to introduce a “normal” amount of carbon into the alloy, which can partly substitute for the expensive alloying element Co, and make the future implementation of the alloy more economically viable. Significant improvement in toughness has been achieved by adding Cu to modify the Laves phase distribution. However, slightly compromised creep strength was found in the newly designed alloys.

Nucleation of Z-phase precipitates under the influence of C

2014-2015

According to our previous results increasing the content of carbon – a cheap austenite stabilizer – led to the formation of various carbides and carbonitrides, such as $M_{23}C_6$, Ta(C, N), together with Z-phase. Z-phase is formed by only one pathway: phase transformation from MX by introducing Cr layers into the cubic lattice of MX. We found that the phase transformation rate from MX to Z-phase is much lower in the steels that contain a high amount of C. In other word, phase transformation from MN to Z-phase is much faster compared to their high C counterpart – M(C,N) [9].

We will systematically change the C content in three test alloys, and use Atom Probe Tomography (APT) and electron energy loss spectroscopy (EELS) to obtain chemical information of the precipitates at the initial stage of precipitation and phase transformation after prolonged exposures at 650 °C. Parallel to our experimental work, first principles calculations of the nucleation process will be carried out by our collaborators through the EU project, Prof. Christian Elsässer at Fraunhofer IWM, Germany. The calculation results will be compared with experimental observations.

Special effects of Ta in combination with Nb

2015-2016

Ta, compared to other Z-phase forming elements, for example Nb, provides a better creep strength to these new steels (Figure 1). The underlying mechanisms were studied in great detail by ATP and transmission electron microscopy (TEM). It has been shown that Ta has the special effect of inducing very fine and densely distributed precipitates [10] into the steel. However, the effect remains largely unknown on combining Ta and Nb to form strengthening Z-phase precipitates. One alloy with combined Ta and Nb addition will be designed. In parallel first principles calculation using density functional theory will be performed, and the key factors concerning the effect of different Z-phase forming elements will be identified.

Effects of B

2016-2017

In 9–12% Cr steels boron can hardly dissolve in the steel matrix; it either dissolves in certain precipitates, such as in $M_{23}C_6$, or forms BN or metal borides. Based on earlier studies on 9–12% Cr steels, B is a very beneficial element in terms of creep, which is believed to connect to its ability of hindering the coarsening of $M_{23}C_6$ [11]. In the Z-phase strengthened steels, we found that B is enriched at interfaces between Z-phase precipitates and the steel matrix [10]. This can influence the interfacial energy of the precipitates, which in turn can affect their nucleation and growth process. However, too much B addition can form large BN, which can consume N, and therefore negatively affects the formation of strengthening Z-phase precipitates. Since B is a light element, which is very difficult to analyse quantitatively in a TEM using techniques like Energy Dispersive X-ray (EDX) spectroscopy or EELS, we plan to use APT to tackle this problem.

Possibility of combining other precipitate families for strengthening

2015-2017

The strengthening effect of Laves phase is a very relevant issue in this case, since compared to all the traditional 9–12% steels, they start to form at the very early stage (within a few hours) at 650 °C. The coarsening rate of this family of precipitates is known to be slower than $M_{23}C_6$. In KME 510, we used Cu to modify Laves phase distribution and thus improved the impact strength of the steel [12]. We will further explore the possibility of using Laves phase to strengthen the steel by investigating Cu addition, and hybrid Laves phase, which contains both W and Mo.

We will fine-tune the C addition (instead of 50 wt. ppm for the first experimental steels) into the steel system. Higher C addition has another beneficial effect, i.e. it can form the precipitate family of $M_{23}C_6$, which is known to be an important category of strengthening particles for almost all the 9–12% Cr steels so far. On the other hand, it introduces carbonitrides, which do not so readily transform to Z-phase. To find the optimal balance will be the focus.

Evolution of microstructure and its influence on the creep mechanisms

2016-2018

Firstly, we plan to use a Scanning Electron Microscope (SEM) equipped with an EDX spectroscopy and an Electron Backscatter Diffraction (EBSD) system to investigate the microstructure evolution of the steels on a relatively large scale. We will track the grain and sub-grain size changes during creep using EBSD. We will use EDX to track the evolution of different precipitates of micrometre-size. We will use energy filtered TEM (EFTEM) to track the evolution of precipitates of sub-micrometre-size. We are going to use APT to directly investigate the number density of the very small precipitates at the initial stages, and also to track the chemical composition changes of the matrix and the precipitates, thus predicting the possible precipitation reactions.

Time schedule

Start the project: 1st September 2014

Finish the project: 15th April 2018

Send in final report: 15th May 2018

Economical account: 15th May 2018

Progress report 1: 1st September 2015

Progress report 2: 1st September 2016

Understanding nucleation of Z-phase precipitates under the influence of carbon: 2014-2015

Understanding special effects of Ta in combination with Nb: 2015-2016

Understanding effects of boron: 2016-2017

Understanding possibility of combining other precipitate families for strengthening: 2015-2017

Understanding evolution of microstructure and its influence on the creep mechanisms: 2016-2018

Staff

Dr. Fang Liu at Chalmers University of Technology, works 30% in this project. During 2014 and 2015 her salary cost is covered by the EU FP7 Z-Ultra programme. Professor Hans-Olof Andrén at Chalmers University of Technology, works 5% in this project (salary also covered by Z-Ultra in 2014 and 2015). Ph.D student Masoud Rashidi works 80% in this project.

Planned Licentiate for Masoud Rashidi: Oct. 2015;

Planned Ph.D defence for Masoud Rashidi: May 2018.

Industrial reference and financing

Siemens Industrial Turbomachinery AB (Lennart Johansson) will participate in the project, and perform in-kind work (tensile testing, impact toughness testing, creep testing, reporting and travel) costing 300,000 SEK per year during 2014-2018. Approximately 25 creep tests can be started, and 12-14 creep machines for testing at 625 and 650 °C will be reserved for the project.

Additional financing from the Research Foundation of VGB will be requested.

Costs (tkr)

Kostnader	2014	2015	2016	2017	2018	Summa
Lönekostnader	137	422	801	856	329	2545
Köpta tjänster						
Utrustning						
Material						
Laboratoriekostnad						
Resor	15	40	40	40	15	150
Övriga kostnader: Mikroskopikostnader	67	200	200	200	67	734
Indirekta kostnader	61	188	356	381	146	1132

Summa kostnader	280	850	1397	1477	557	4561
Summa sökta medel	200	200	200	200	0	800

The total budget is calculated based on the real cost for the project: salary + instrument users' fee + travel + indirect cost. The already existing finance sources: Energimyndigheten (until 2015) and EU (until 2015) have been taken into account. More resources will be applied via VGB research foundation in Germany.

Salary:

4 months in 2014, a full year of 2015, 2016, 2017, and 4.5 months in 2018.

Ph.D student: 80%.

Fang Liu: 30%. **Note:** this part comes in from 2016, since it has been covered by the EU project until the end of 2015.

Hans-Olof Andrén: 5%. **Note:** this part comes in from 2016, since it has been covered by the EU project until the end of 2015.

Instrument cost

This project largely based on using different advanced microscopy and microanalysis techniques. The instrument operation cost is estimated as about 200 tkr per year. It can vary a little year to year, but the cost is at this level in average.

Travel

Outside of Europe

In average there are 1 to 2 important international conferences outside of Europe per year, and each costs roughly 20 tkr. It is about 30 tkr annually.

Short travels

There are 4 travels to collaboration partners within Sweden, or to Denmark. Counting in train tickets and one overnight, each trip costs about 2.5 tkr. It is about 10 tkr per year.

The total travel cost adds up to 40 tkr per year.

Indirect cost

Overhead at Chalmers is at present 44.5% on salary cost.

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Bilaga till projektansökan inom Samverkansprogrammet Materialteknik för t

Fördelning av kostnader för varje ingående part

Sökande:	Chalmers teniska högskola
Projekttitel:	KME-710 Design av en ny generation 12% kromstål

Part		Kostnadsfördelning									
		Lön	Konsult	Utrustning	Material	Laboratorie	Dator	Resor	Övrigt	Indirekta	Summa
Chalmers	2014	98						10	48	44	200
	2015	99						10	47	44	200
	2016	115						6	28	51	200
	2017	116						6	27	51	200
	2018										0
	Summa	428	0	0	0	0	0	32	150	190	800
Siemens	2014	100				195		5			300
	2015	100				195		5			300
	2016	100				195		5			300
	2017	100				195		5			300
	2018										0
	Summa	400	0	0	0	780	0	20	0	0	1 200

STÖD FÖR REGISTRERING I ENERGIMYNDIGHETENS WEBBPORTAL

Följande siffror kan användas för att fylla i projektets ansökan på Energimyndighetens webbportal E-kanalen. Tabellen skapas automatiskt utifrån

	SUMMA	2014	2015	2016	2017	2018
Lönekostnad	828	198	199	215	216	0
Konsultkostnad	0	0	0	0	0	0
Utrustning	0	0	0	0	0	0
Material	0	0	0	0	0	0
Laboratoriekostnad	780	195	195	195	195	0
Datorkostnad	0	0	0	0	0	0
Resor	52	15	15	11	11	0
Övrigt	150	48	47	28	27	0
Indirekta kostnader	190	44	44	51	51	0
SUMMA	2 000	500	500	500	500	0